THE EFFECT OF CARBONATION ON CREEP AND SHRINKAGE OF PORTLAND CEMENT CONCRETE

by

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D E D I C A T I O N

To Mr. Hermand E. Monserrate: for his inspiration and guidance.

ABSTRACT

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Francisco Orlando Martin Alvarez

Submitted to the Course in Building Engineering and Construction of the Department of Civil and Sanitary Engineering on May 20, 1961 in partial fulfillment of the requirements for the degree of Master of Science.

The present study consists of determining how the creep and shrinkage of concrete is affected by various amounts of CO_2 and moisture in the surrounding atmosphere. Both loaded and unloaded specimens were stored in controlled humidities of 33, 52, 76, and 97 percent. Four different percentages of CO_2 were associated with each relative humidity: 0, .03, 50, and 100 percent, giving a total of 16 different storage conditions.

The data collected indicated that the carbonation shrinkage tends to be higher in atmospheres containing higher percentage of carbonic gas. The effect of humidity on carbonation tended to be extremely important, with 52 percent relative humidity producing the highest carbonation shrinkage. In addition, it was found that the rate of carbonation was highest during the earliest stages of the carbonation process.

An attempt was made to measure the influence of carbonation on creep. No conclusive evidence was found that carbonation reduces creep. The results showed that carbonation shrinkage of the loaded specimens was slightly lower due to partly closing of the pores under load.

The collected data is portrayed graphically as well as in tabular form, photographs of the pertinent apparatus are included to illustrate experimental techniques. Detailed information concerning the general testing methods is presented.

Thesis Supervisor: J. Lloyd Cutcliffe

Title: Instructor

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INTRODUCTION

General Concrete Construction

Concrete is a mixture of inert materials of varying sizes, which are bound together with a cement paste. The whole mass is deposited in a plastic or fluid state; and, almost immediately after placing, a hardening process start which, under the proper curing conditions, may continue for years. Because it is placed in a plastic condition, concrete lends itself successfully to all kinds of construction; provided suitable molds are used or special placing equipment is available, such as a tremie[1] for underwater work placing.

Concrete possesses certain characteristics that have been responsible for its great demand as a building material. Since concrete is a physically amorphous material (after mixing) it can be easily handled in common types of jobs as well as being adaptable to complex shapes. This material provides excellent strength for structural uses. High strength developed by modern concretes enables the designer to employ various methods of design, e.g., prestressed concrete design. Although it is a high strength material, it has a relatively low cost; one of the many reasons why it has been used extensively.

Durability, low maintenance cost, fire resistance are but a few of the practical qualities responsible for its broad acceptance as a major construction material. In addition to the practicability

of the material, structures of concrete exhibit many aesthetic qualities.

Concrete Behavior

A structural unit may be considered a failure when the purpose for which it was designed is unaccomplished. Failures can be divided into two general categories. These are: First, when the structure completely or partially collapses due to gravitational, earthquake, wind, as well as yielding of the supports. Second, when the structure can still hold the imposed loads but deflections and cracks make it unsuitable to accomplish the purpose for which it was designed. Both types of failure are directly related to the stresses in the concrete set up by the loads and volume changes, such as, thermal contraction and expansion, shrinkage and creep.

The effect of loads on structures has been extensively investigated by a large number of authors as to different types of loading techniques as to determine the behavior of the concrete structures. Laboratory test samples as well as completed units have been load tested to determine the different stress patterns set up by these forces.

Temperature effects[2] have been the cause of failure of a great many number of structures. These effects have also been studied to determine the mechanism of expansion and contraction on concrete. The results of the aforesaid studies have been applied to a large number of commercial thermal expansion joints to prevent these failures.

Most of the previous work on thermal changes of concrete has determined that temperature changes may occur at any time and continue indefinitely or significant temperature changes may occur over short periods and may be repeated frequently.[3] Other factors that are connected with temperature changes in length:

(1) Magnitude of the change

(2) Rate of change and distribution

(3) Coefficient of expansion of the concrete

(4) Modulus of elasticity of the concrete

(5) The degree of restraint

(6) Coefficient of expansion of the reinforcement and aggregates

(7) Type of cement

Concretes under load tend to creep and shrink. Creep or plastic flow is defined as increased deformation with time at constant load. Shrinkage is usually associated with a change in length due to water loss of the member. Under ordinary conditions shrinkage and creep have the same direction even though they may have different magnitudes.

Plastic flow or creep is due largely to the seepage of water from the gel when an external load is applied to the concrete. The flow of water to or from the cement gel takes place through minute gel pores. The flow through the gel pores is a function of the pressure gradient, thus resulting in a more rapid expulsion of water. Powers[4]

has explained this as an entropy effect. The rate of expulsion is also a function of the vapor pressure on the outside of the mass. Shrinkage caused by the loss of moisture, as well as time yield due to seepage, are interrelated phenomena, although they may be conveniently considered as separate and additive in their effects.

Davis[5] suggested that differences in flow observed in concrete containing different mineral aggregates may be due to crystalline flow. He also suggested that the existence of lateral flow, accompanying axial flow when the lateral dimensions are unrestrained, may be caused by viscous flow.

Presently there are many theories related to this phenomena and some are extensions, with external stress introduced as a variable, of shrinkage. A typical curve is shown in Figure (1) for a concrete in compression under a load of 600 pounds per square inch. From this figure the creep is seen to occur rapidly in the first few weeks after loading and then to proceed at a steadily diminishing rate. The creep is directly proportional to the applied stress up to a high proportion of the strength and this holds even down to the smallest loads for which measurements are practicable.

Davis found that the higher the sustained stress, the greater the flow. The moisture content of the cement gel has a marked effect upon the time yield; the flow of a totally dry concrete being of small magnitude. The extent of hydration of the cement alters the time vs. flow relation appreciably, since concrete of greater age under given curing conditions at the time of loading shows less plastic flow. In

plain concrete flow has been observed to continue even after almost 7 years under sustained stress, although the rate of flow is very small at such ages. Usually the flow practically ceased after 1 or 2 years.

Ostlund[6] has developed mathematical expressions taking into consideration various factors; viz., the magnitude of the stress, the time which has elapsed since the load was applied, the age of the concrete when the load was applied, the dimensions of the test body, the composition of the concrete, the atmosphere around the sample, the components of the concrete and so on. It is known from experiments that creep, as well as the rate of creep, increases when the atmosphere around the test body becomes drier. Also the rate of creep decreases with increasing dimensions of the test body.

Small plain concrete cylinders stored in dry air under sustained stress (within the range of ordinary working stresses for concrete) may have a total change in length, due to flow and shrinkage combined, equivalent to 1 or 2 inches per 100 feet in 7 years. For cylinders stored under moisture conditions, the total deformation due to flow and expansion may be only one-fourth to one-third of the total deformation of similar concretes stored in dry air.

Davis found the total unit time deformation of air-stored reinforced concrete columns to be from one-third to one-half of those in corresponding air-stored plain concrete columns. These deformations cause appreciable changes in the distribution of stress between the

steel and concrete in reinforced concrete columns under load in dry air. Under such deformations the stress in the steel is increased.

Washa[7] reported on plastic flow of thin reinforced concrete slabs of a given span, water cement ratio and curing method. The greatest creep deflections were obtained for the slabs made with the largest water content. This result was most pronounced for the longest span, and least pronounced for the shortest span. Some beams were sealed by painting them with Bakelite lacquer and then with paraffin. Plastic flow, shrinkage, warpage and weight loss all took place at a slower rate for the sealed beams than for the dry (unsealed) specimens. The ultimate shrinkage and weight loss were about the same for the two curing conditions, but the sealed specimens had higher modulus of elasticity and ultimate compressive strengths at 5 years. The sealed specimens generally had lower plastic flow deflections and deformations.

Shrinkage has generally been attributed simply to water loss of the mass but present evidence indicates the exact mechanism of shrinkage is far more complicated than this explanation indicates. Some theories related to the capillary structure of the material and the nature of the binding agent postulate such things as capillary tension, surface sorption, swelling pressure of a gel and interlayer of crystals as possible explanation of shrinkage. The capillary tension theory[8] shrinkage is associated with the increase of the tension at the water meniscus in the capillaries as drying proceeds. The theory is based on Kelvin's equation that there is a certain

radius of curvature of a water meniscus that corresponds to a given reduction in vapor pressure. As water recedes in the capillaries this curvature increases and the tension forces produce a tension in the water remaining in the capillary pores. This liquid tension must be balanced by an equivalent compressive stress in the solid producing shrinkage deformation. When water is taken up the meniscus advances up the capillary, the radius of curvature decreases and finally vanishes when free water is present at the surface. During this process the liquid tension and induced compressive stress and shrinkage lessen and finally disappear. The surface sorption theories are associated with the cement gel itself since it is here that the greak bulk of the surface area lies. We have seen from former investigators that the gel can absorb water and that the amount of it held in films on the internal surface depends on the vapor pressure. This film of water is held between the particles of the gel; the thickness of the film determining the separation of the particles. Therefore shrinkage will be suspected. The swelling pressure of the gel theory takes into consideration that cement sets in the fully saturated condition with its gel fully expanded. As it dries and water leaves the gel, negative pressures or tensions will be set up inside it. The resulting contraction is resisted by the rigid framework of the mass, which is thus put into tension. This internal tension could be called the cause of shrinkage. The interlayer of crystals theory takes into consideration changes in the lattice spacing of the hydrated calcium silicate crystals with their water content. These crystals have a

layer structure and water molecules can enter between layers expanding thus the lattice. Evidence of a similar effect with hydrated calcium tetra-aluminate has also been found by an overall movement of the solid. This accounts for some dimensional changes in concrete.

No conclusion can be drawn from these shrinkage mechanisms but it seems that capillary effects may operate at high relative humidities, e.g., 70-80 percent and above; gel sorption at intermediate humidities and the lattice shrinkage of hydrated calcium silicate at low humidities. It must be noted that when set cement or concrete loses water, the change in volume is only a small fraction of the volume of the water lost. A typical shrinkage curve is shown in Figure (2) at constant atmosphere conditions.

The following are the most important factors that influence drying shrinkage:[9][10][11] the water cement ratio of the paste, the composition and fineness of the cement, the amount of paste in the concrete, the characteristics and amount of admixtures used, the mix proportions, the mineral composition of the aggregates, the maximum size of aggregates, the size and shape of the concrete mass, the amount and distribution of reinforcement, the curing conditions, the length of the drying period, the humidity of the surrounding air, cement and water content, gradation of aggregates, age at first observation, duration of test and absorptiveness of form. These factors are also associated with the phenomenon of creep. This association is why some authors have described creep as a special case of shrinkage under load.

All of the previously mentioned factors have a definite bearing on the problem of shrinkage and creep. There are some other problems that have not been studied which may have a definite bearing on creep. The purpose of this study consists of determining the effect of different carbonic gas concentrations on shrinkage while at the same time extending such investigation to creep flow.

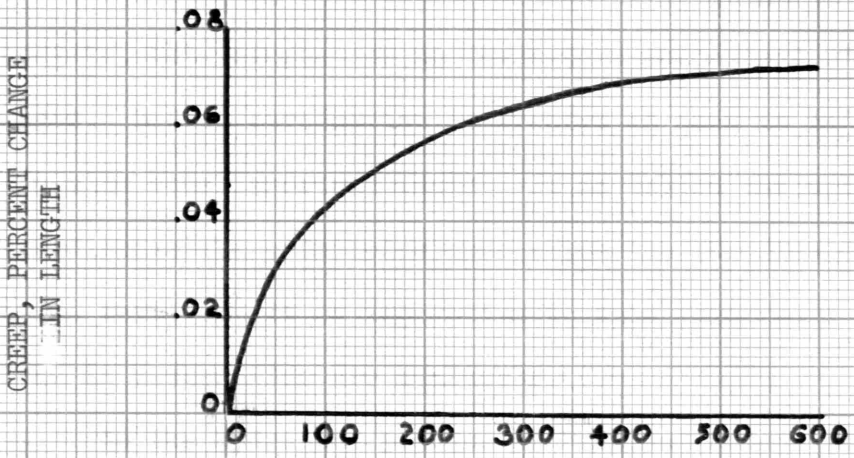

Figure 1

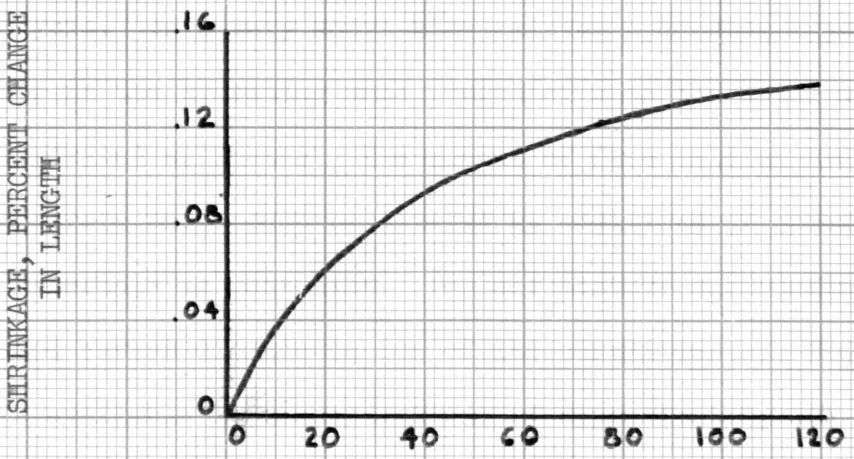

Figure 2

II. PREVIOUS AND PRESENT WORK ON CARBONATION

Nature of Carbonation

It has long been accepted that carbonic gas has an effect on concrete, but information on the nature and extent of this effect is meager. Before carbonation is treated as such it is desirable to have in mind some important facts. It is beyond the scope of this work to discuss the principles upon which the theories of cement hydration rest; but it is imperative to know from where the compounds involved in carbonation are available.

In the hydration process of Portland cement it is well agreed that "tricalcium aluminate reacts extremely rapidly and the tetracalcium aluminoferrite very quickly."* Attack of tricalcium silicate soon leads to saturation of the solution with lime due to hydrolysis. In a very short time calcium hydroxide crystals can develop in the hydrating mass, together with the usual structure less hydrated silicates. The amount of free calcium hydroxide in the concrete mass could be altered by additions or combinations with such materials as pozzolans. The air mixture surrounding the concrete contains carbon dioxide as one of its constituents. Therefore, this gas is available in concentrations of approximately .03 per cent (.0003 atmosphere).

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*Chemistry of Cement and Concrete, Lea and Desch, 1955, p. 205.

Toennies[12] has defined carbonation as the chemical combining of carbon dioxide and the hydration product of portland cement. The primary importance in this chemical reaction is the combination of calcium hydroxide with carbonic gas, in the presence of water, to yield calcium carbonate and more water, e.g.

$$Ca(OH)_2 + CO_2 = CaCO_3 + H_2O$$

In the case of lime plasters and mortars Voss[13] considers that this reaction still follows through in some cases and in the presence of excess CO_2 the following reaction takes place:

$$CaCO_3 + CO_2 + H_2O = Ca(HCO_3)_2$$

This new compound tends to close small fissures and cracks; thus the term autogenous healing has been coined.

Although there exist experimental evidence that substantially all of the constituents of cement are subject to ultimate carbonation under ideal conditions, the author considers that under ordinary service conditions of the material the most important reason is the combination of free lime ($Ca(OH)_2$) with carbonic gas (CO_2).

The work of Bessey[14] shows some light in higher lime content mixes of cement

Previous Works on Carbonation

A great many investigators have realized that the combination of carbon dioxide with the cement components is a practical and

important problem. Some of these men will be mentioned at this stage of this report.

Shiedeler[15] realized that carbonation of masonry units is accompanied by their linear shrinkage. He also found that the moisture condition of the masonry unit has had an influence on their receptiveness. Units predried to a moisture condition in equilibrium with air at 50 percent relative humidity could be artificially carbonated, while those predried at 11 percent could not. This has been verified by the more recent work of Verbeck[16], who found that mortar specimens would carbonate less at 25 percent relative humidity than at 50 or 75 percent. Verbeck also reported little carbonation with specimens exposed to a relative humidity of 100 percent. This confirmed the work of Leber and Blakey[17] who found that artificial carbonation was retarded when the specimens were exposed to carbon dioxide, while in a wet condition. Thus it would appear that the optimum moisture condition for artificial carbonation is somewhere between the wet and dry state.

Based on these principles, Toennies derived machines to develop a process of artificial carbonation of concrete masonry units. This process of hastening carbonation or preshrinking would eliminate subsequent atmospheric carbonation and attendant shrinkage after the units were in the wall. Research was prompted by the feasibility of artificial carbonating concrete masonry units using as a source of carbon dioxide the flue gases from combustion in steam boilers adjunct to steam curing.

Brady[18] calculated from the density of crystalline calcium hydroxide and calcite that the effect of carbonation in a 1:3 cement/sand mortar should be an increase in volume of about .05 percent as the calcium hydroxide is converted to calcium carbonate. However, an experimental study showed an increase in shrinkage after carbon dioxide treatment. The specimens were left for 7 days in moist air and then partly dried out in an atmosphere of 63.5 percent relative humidity and 20°C for 3 days. Some were kept in carbon dioxide and others in air at the same relative humidity and temperature. The specimens stored in air showed only a slight shrinkage, whereas specimens stored in carbon dioxide showed a further shrinkage of the order of two-thirds of the initial shrinkage.

Lea and Desch[19] referring to the action of carbon dioxide commented that this gas in tunnels may be present in relatively large quantities in the atmosphere, but when the tunnel is fairly dry it produces no ill effects. But under moist condition, however, fairly rapid attack of concrete lining of tunnels or mortar pointings to brickwork is occasionally experienced. In water-saturated atmosphere a skin of calcium carbonate is formed on the wet surface (Bessey), preventing the penetration of carbon dioxide. Carbonation of concrete, both in the field and test specimens, is therefore to be expected more under semi-dry conditions.

The time when the carbonation takes place seems to be important. Mansfield[20] claimed that carbonation is detrimental

when it occurs before the portland cement hydration compounds are formed, but is beneficial when it follows hydration.

Meyers[21] investigated the effect of carbon dioxide on the strength and shrinkage of mortars composed of one part cement to four parts of sand. He cured all specimens in water for 7 days and then stored them in sealed containers. A carbon dioxide absorbent was placed in the container with those specimens to be used as control. The pore water was removed by vacuum from the remaining specimens and they were kept in carbon dioxide for 10 days at 50 psi. Some of these specimens were further treated for 15 days and others for a month with carbon dioxide at 150 psi. It was shown that shrinkage of the specimens treated with carbon dioxide at 150 psi for one month was only 30 to 40 percent of that for untreated specimens, and attained considerably higher strength.

Kauer and Freeman[22] subjected fresh concrete specimens to carbon dioxide immediately after molding. These specimens were then cured in various humidities and temperatures in CO_2 atmospheres ranging from 4.5 to 18 percent for 24 to 96 hours. Tests were then made on hardness of surface, depth of carbonation, amount of carbonation on surface as compared with unexposed specimens and compressive strength. These tests indicated that fresh concrete exposed to CO_2 resulting from heating devices which exhaust the flue gases directly into the room, have soft surface of various depth depending on the concentration of CO_2, the temperature at which concrete is cured.

Leber and Blakey experiments have concluded that:

(1) Carbon dioxide can have either a detrimental or beneficial effect on shrinkage of mortars or concretes.

(2) The time the samples are exposed to carbon dioxide is important. The first 10 days seem to be critical for all changes that will develop later.

(3) Carbon dioxide apparently not only reacts with the calcium hydroxide resulting from the hydration of mortars and concretes but decomposes other constituents, forming calcium carbonate.

Weber and Mather[23] exposed small slabs of cast stone, 6 by 4 by 1 inch thick to the action of carbonic gas at different pressures (50, 100 and 150 psi). The samples in this test were of various ages freshly molded, 24 hours, 48 hours, 1 week and 2 weeks. After treatment with CO_2 the freshly molded concrete samples were chalky and crumbly, most probably because the water of the mixture has formed carbonic acid and precipitated the lime and calcium carbonate before the complete reaction of the silicates. They found also that the higher the pressure, the more total gas was absorbed; but that seemed to make little difference insofar as surface hardness was concerned.

Powers and Brownyard,[24] in determining some physical properties of hardened portland cement, found that carbonation was off-shooting their results. They agree that at early ages of

hydration the relative error would be considerably greater for the same amount of carbonation at later ages.

Lea and Desch have suggested that the phenomenon of crazing in concrete has been due to carbonation of the enriched skin formed in the concrete surface due to excessive trowelling fringing up the cement to the surface. Crazing consists of a network of fine hair cracks over the surface of the material. Generally it is not considered a major problem except where the final surface is of prime decorative importance.

The proper use of concrete as a structural material requires knowledge of all the properties which influence its performance. Data on volume change properties such as drying shrinkage and creep under sustained stress are of considerable importance to designers and producers of concrete structures and elements. This is particularly important in the field of prestressed and masonry unit construction. The previous mentioned authors' works present the results of comprehensive laboratory tests of concretes typical of those being used in various applications of carbonation.

The Present Work

The present study is concerned with the relative action of carbon dioxide in creep flow of concrete. Many factors, such as relative humidity during exposure to carbon dioxide, carbon dioxide concentration, and specimen size are of considerable importance to both the rate and degree to which carbonation occurs. The magnitude

of the shrinkage and creep that is directly produced during carbonation and the volume stability of the carbonated products are of particular concern.

The use of mortars permits the fabrication of small specimens from which better observation of the details of the studied mechanism can be made than would be possible with larger size specimens required for concrete. Therefore the results obtained will be qualitatively applicable to concretes.

It has been well-known that the moisture content or relative humidity of the hydrated paste has a considerable and important influence on the rate of ultimate extent of carbonation. A clear understanding of this aspect is valuable in the interpretation of various observations regarding carbonation and also because of its theoretical implications.

As mentioned before, specimen size can influence the carbonation process. It can influence the apparent relationship between relative humidity and carbonation; if carbonation proceeds at a relative rapid rate. This effect, which will be discussed later, is brought about by release of moisture within the specimen by carbonation. If the specimen is small or if carbonation proceeds slowly (due to low concentration of CO_2) this moisture can be diffused out of the specimen, causing water loss shrinkage and maintaining the internal relative humidity of the specimen approximately in equilibrium with the internal controlled humidity.

For the purpose of investigating shrinkage and creep due to carbonation, small mortar prisms were prepared using a laboratory blend of equal parts of four locally available portland cements and standard Ottawa sand.

The sand cement and water cement ratio, as well as detailed information of the technique and procedure involved in testing, is given in the Appendix of this report. The specimens were moist cured for 7 days at 73°F. After 7 days, specimens were subject to storage in air free of CO_2 at 97, 76, 52 and 33 percent relative humidity at 73°F. These exposures were continued until all specimens had reached apparent equilibrium, both as to weight and length, at various relative humidities. After moisture equilibrium had been reached, which was assumed to require 85 days, half of the specimens were compressed with approximately 700 pounds per square inch stress applied by a spring mechanism; the remaining half was kept unloaded. All samples were exposed to carbonic gas at different concentrations and at the same relative humidity that the samples were stabilized originally. The pressure of the surrounding atmosphere was maintained constant (14.7 psi). At the end of the carbonation period, which required 90 days, the samples were removed from the dioxide atmosphere and subsequently the results will be shown.

III. RESULTS

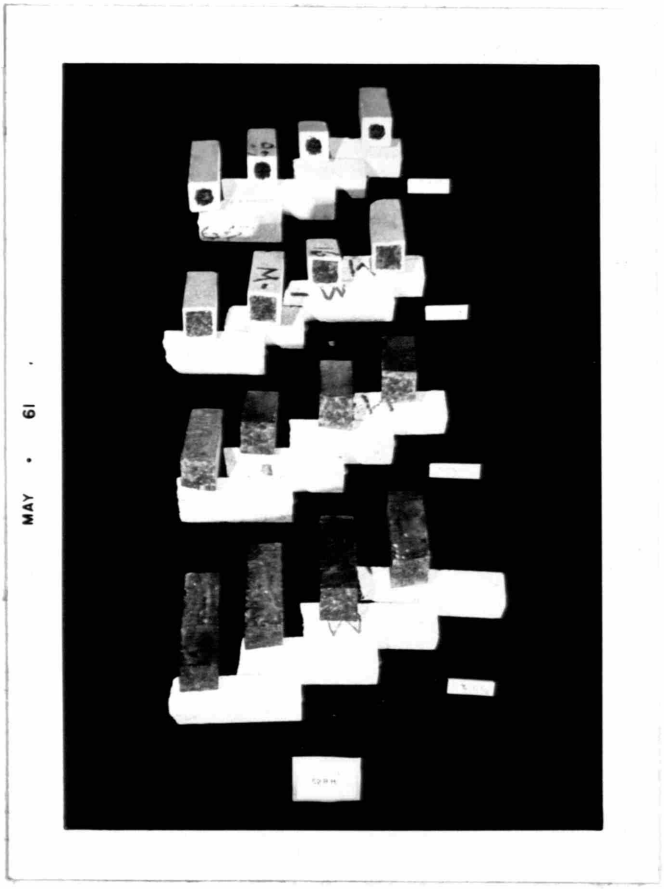
Photographic Data

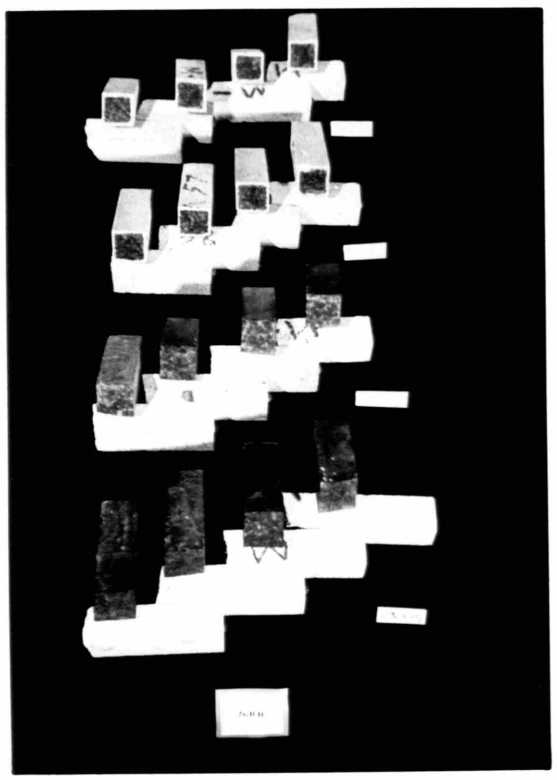
Photograph 1

DEPTH OF CARBONATION OF
SAMPLES EXPOSED TO 33% R.H.

Photograph 2

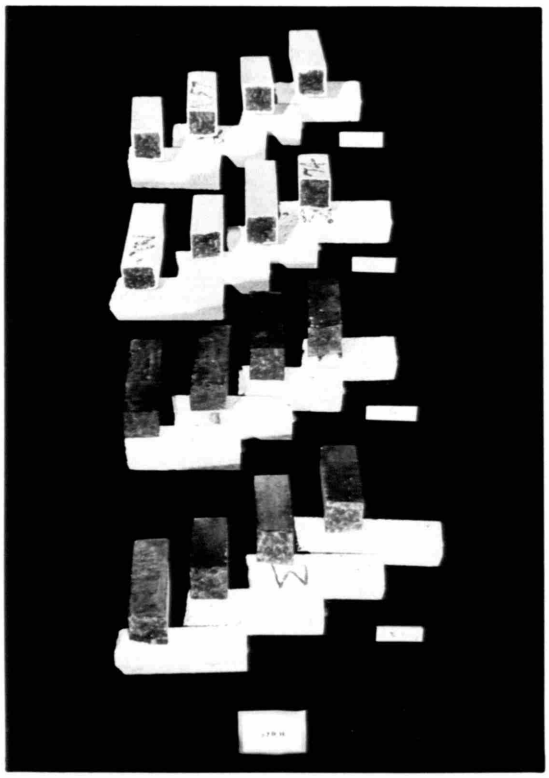
DEPTH OF CARBONATION OF
SAMPLES EXPOSED TO 52% R.H.





Photograph 3

DEPTH OF CARBONATION OF
SAMPLES EXPOSED TO 76% R.H.



Photograph 4

DEPTH OF CARBONATION OF
SAMPLES EXPOSED TO 97% R.H.

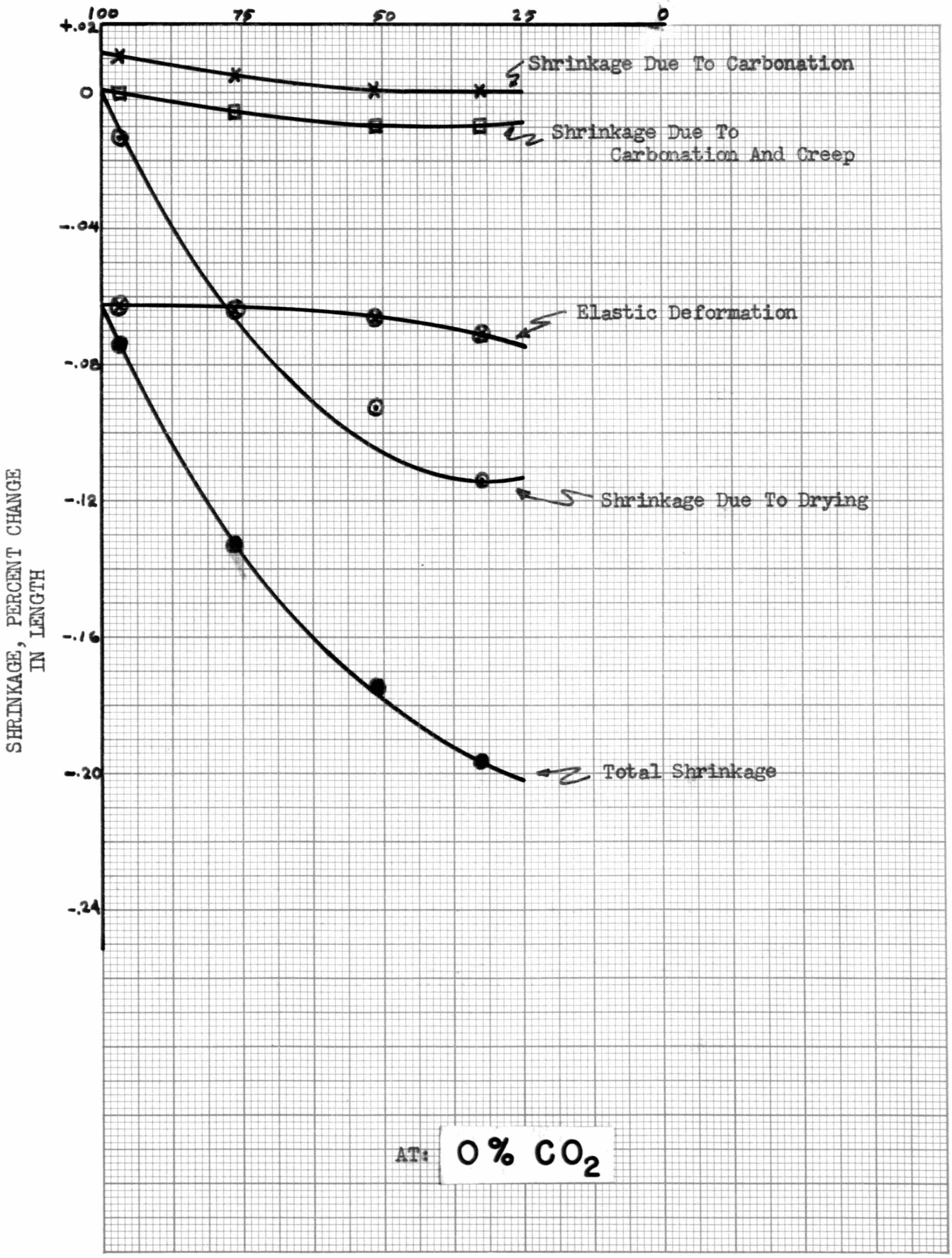
TABLE I

0% CO_2

LENGTH AND WEIGHT CHANGE AT RELATIVE HUMIDITIES SHOWN

Nominal Sample: 1 by 1 by $6\frac{1}{4}$ in.

	97% R.H.	76% R.H.	52% R.H.	33% R.H.
Length Change, percent				
During CO_2-free air drying (85 days)	-0.015	-0.067	-0.092	-0.117
During subsequent CO_2 exposure (90 days)	-0.000	-0.004	-0.010	-0.010
Sub-total	-0.015	-0.071	-0.102	-0.127
Due to Loading	-0.062	-0.062	-0.066	-0.070
TOTAL	-0.077	-0.133	-0.168	-0.197
Weight Change, percent				
During CO_2-free air drying	-1.35	-4.31	-5.84	-6.39
During subsequent CO_2 exposure	+0.35	+0.61	+0.67	+0.59
TOTAL	-1.00	-3.70	-5.17	-5.80
Length Change, percent				
Carbonation Shrinkage (percent)	+0.011	+0.007	0.000	-0.002
Creep Flow (percent)	-0.011	-0.011	-0.010	-0.010


RELATIVE HUMIDITY, PERCENT
100
75
50
25
0
+.02
0
-.04
-.08
-.12
-.16
-.20
-.24
SHRINKAGE, PERCENT CHANGE IN LENGTH
Shrinkage Due To Carbonation
Shrinkage Due To Carbonation And Creep
Elastic Deformation
Shrinkage Due To Drying
Total Shrinkage
AT: 0% CO_2

Graph I

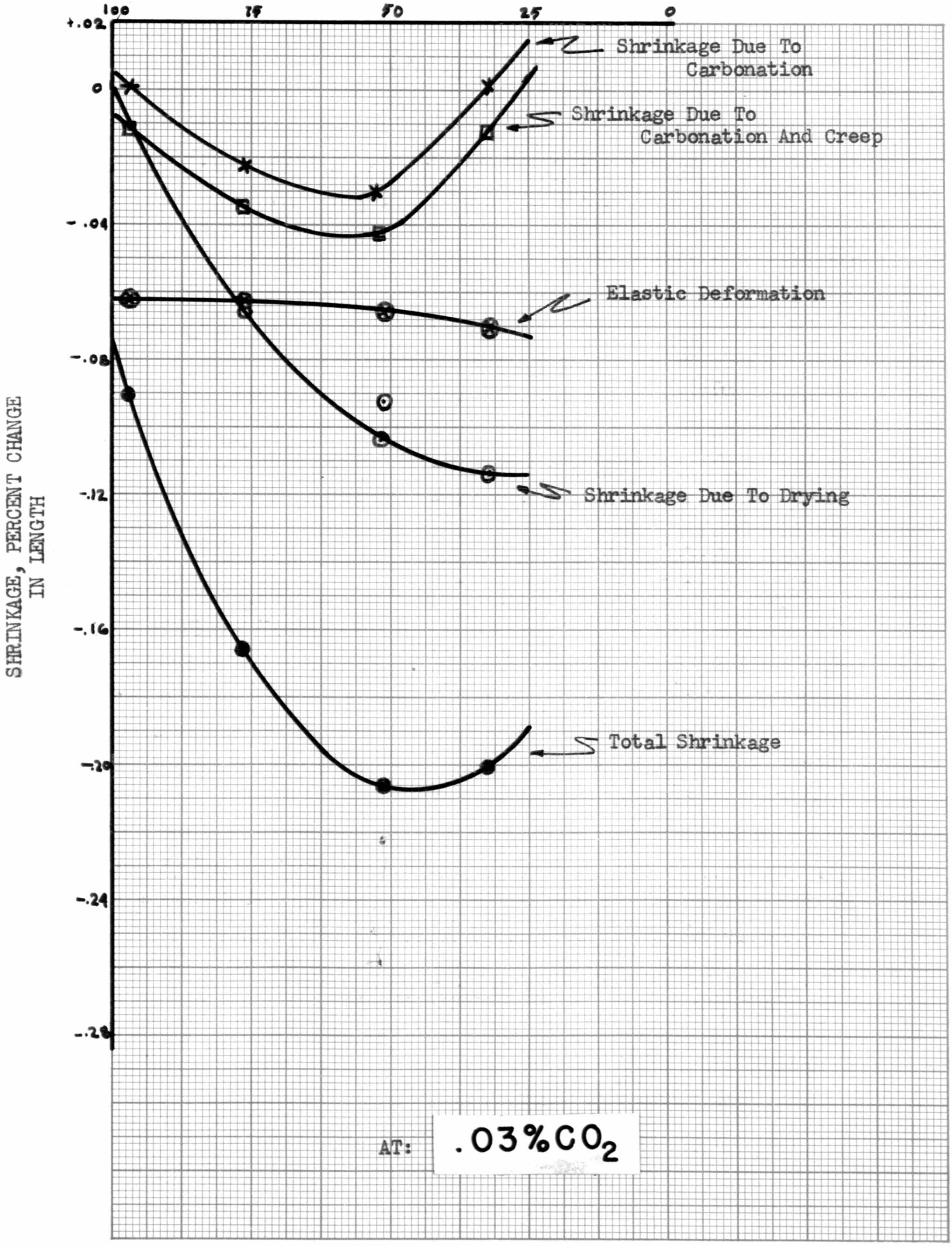
TABLE II

.03% CO_2

LENGTH AND WEIGHT CHANGE AT RELATIVE HUMIDITIES SHOWN

Nominal Sample: 1 by 1 by $6\frac{1}{4}$ in.

	97% R.H.	76% R.H.	52% R.H.	33% R.H.
Length Change, percent				
During CO_2-free air drying (85 days)	-0.015	-0.067	-0.092	-0.117
During subsequent CO_2 exposure (90 days)	-0.012	-0.035	-0.044	-0.012
Sub-total	-0.027	-0.102	-0.136	-0.129
Due to Loading	-0.062	-0.062	-0.066	-0.070
TOTAL	-0.089	-0.164	-0.202	-0.199
Weight Change, percent				
During CO_2-free air drying	-1.35	-4.31	-5.84	-6.39
During subsequent CO_2 exposure	+0.68	+0.87	+1.09	+0.60
TOTAL	-0.67	-3.44	-4.75	-5.79
Length Change, percent				
Carbonation Shrinkage (percent)	0.000	-0.024	-0.032	0.000
Creep Flow (percent)	-0.012	-0.011	-0.012	-0.012


RELATIVE HUMIDITY, PERCENT
100
75
50
25
0
+.02
0
-.04
-.08
-.12
-.16
-.20
-.24
-.28
SHRINKAGE, PERCENT CHANGE IN LENGTH
Shrinkage Due To Carbonation
Shrinkage Due To Carbonation And Creep
Elastic Deformation
Shrinkage Due To Drying
Total Shrinkage
AT: .03%CO2

Graph II

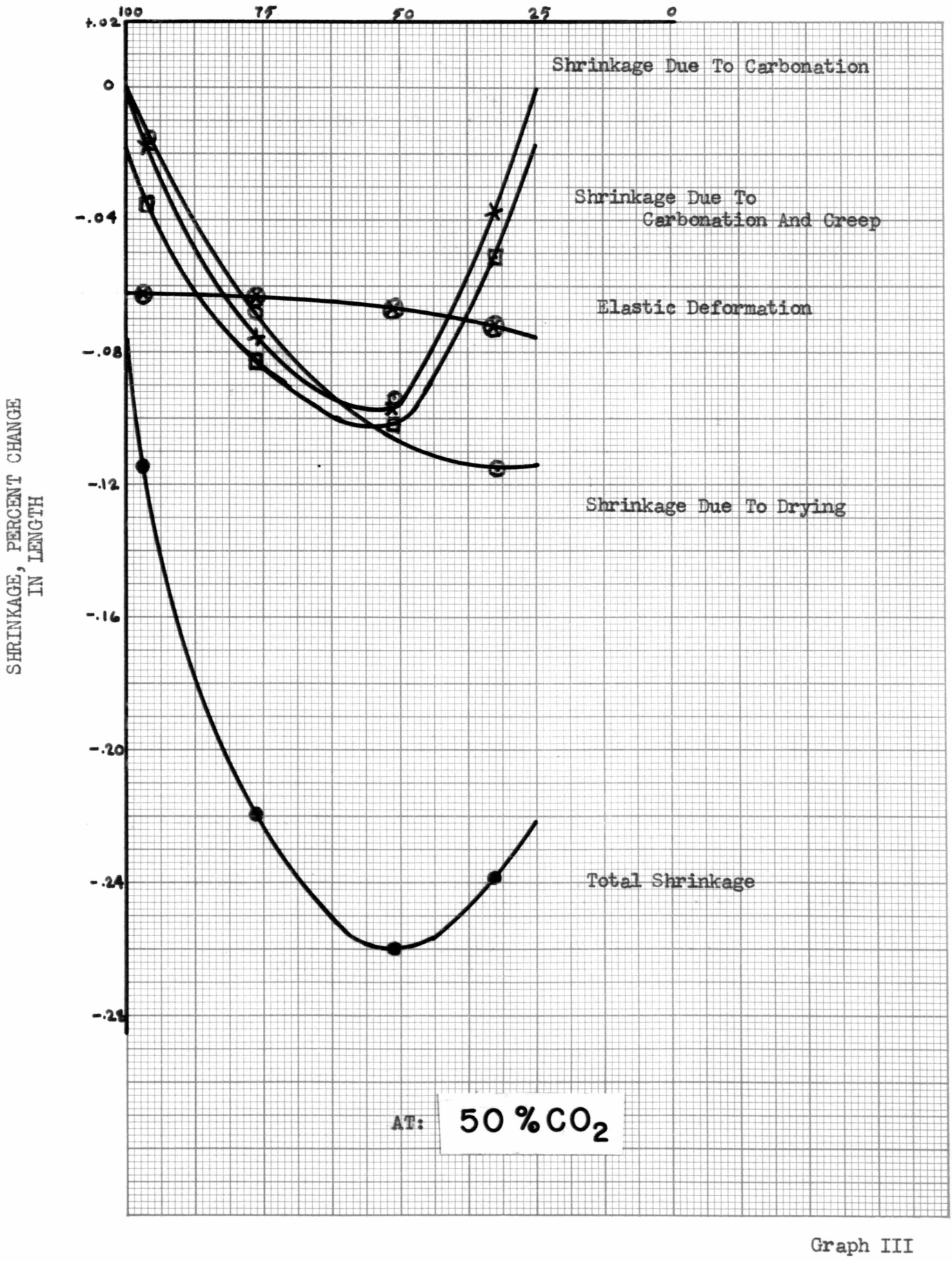
TABLE III

50% CO_2

LENGTH AND WEIGHT CHANGE AT RELATIVE HUMIDITIES SHOWN

Nominal Sample: 1 by 1 by $6\frac{1}{4}$ in.

	97% R.H.	76% R.H.	52% R.H.	33% R.H.
Length Change, percent				
During CO_2-free air drying (85 days)	-0.015	-0.067	-0.092	-0.117
During subsequent CO_2 exposure (90 days)	-0.034	-0.082	-0.103	-0.049
Sub-total	-0.049	-0.149	-0.195	-0.166
Due to Loading	-0.062	-0.062	-0.066	-0.070
TOTAL	-0.111	-0.211	-0.261	-0.236
Weight Change, percent				
During CO_2-free air drying	-1.35	-4.31	-5.84	-6.39
During subsequent CO_2 exposure	+0.87	+2.33	+3.29	+0.66
TOTAL	-0.48	-1.98	-2.55	-5.73
Length Change, percent				
Carbonation Shrinkage (percent)	-0.024	-0.074	-0.098	-0.038
Creep Flow (percent)	-0.010	-0.008	-0.005	-0.011


RELATIVE HUMIDITY, PERCENT
100
75
50
25
0
+.02
0
-.04
-.08
-.12
-.16
-.20
-.24
-.28
SHRINKAGE, PERCENT CHANGE IN LENGTH
Shrinkage Due To Carbonation
Shrinkage Due To Carbonation And Creep
Elastic Deformation
Shrinkage Due To Drying
Total Shrinkage
AT: 50 % CO_2

Graph III

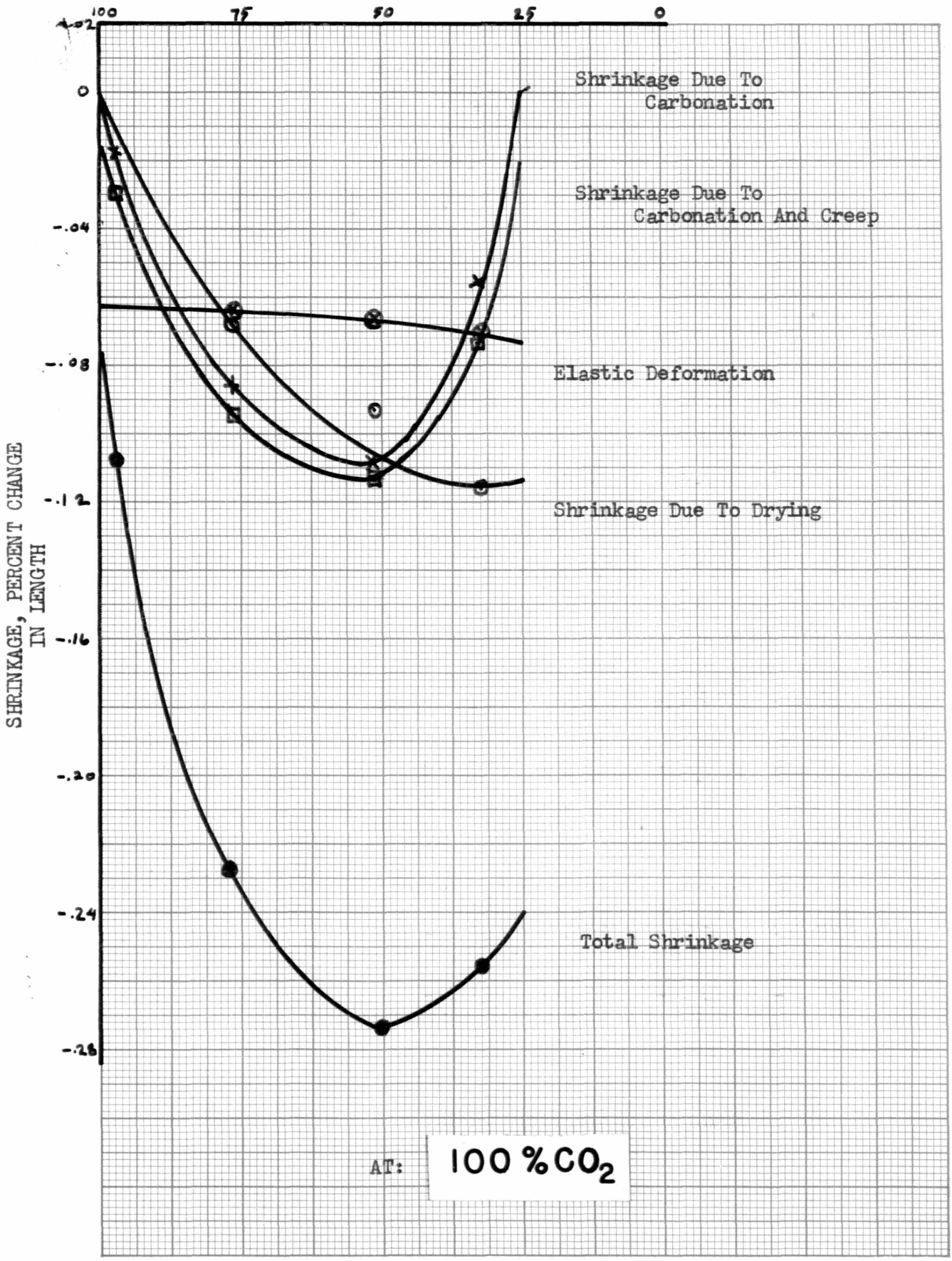
TABLE IV

100% CO_2

LENGTH AND WEIGHT CHANGE AT RELATIVE HUMIDITIES SHOWN

Nominal Sample: 1 by 1 by $6\frac{1}{4}$ in.

	97% R.H.	76% R.H.	52% R.H.	33% R.H.
Length Change, percent				
During CO_2-free air drying (85 days)	-0.015	-0.067	-0.092	-0.117
During subsequent CO_2 exposure (90 days)	-0.029	-0.095	-0.116	-0.067
Sub-total	-0.044	-0.162	-0.208	-0.184
Due to Loading	-0.062	-0.062	-0.066	-0.070
TOTAL	-0.106	-0.224	-0.274	-0.254
Weight Change, percent				
During CO_2-free air drying	-1.35	-4.31	-5.84	-6.39
During subsequent CO_2 exposure	+1.54	+3.20	+4.59	+1.19
TOTAL	+0.19	-1.11	-1.25	-5.20
Length Change, percent				
Carbonation Shrinkage (percent)	-0.016	-0.085	-0.110	-0.055
Creep Flow (percent)	-0.013	-0.010	-0.006	-0.012

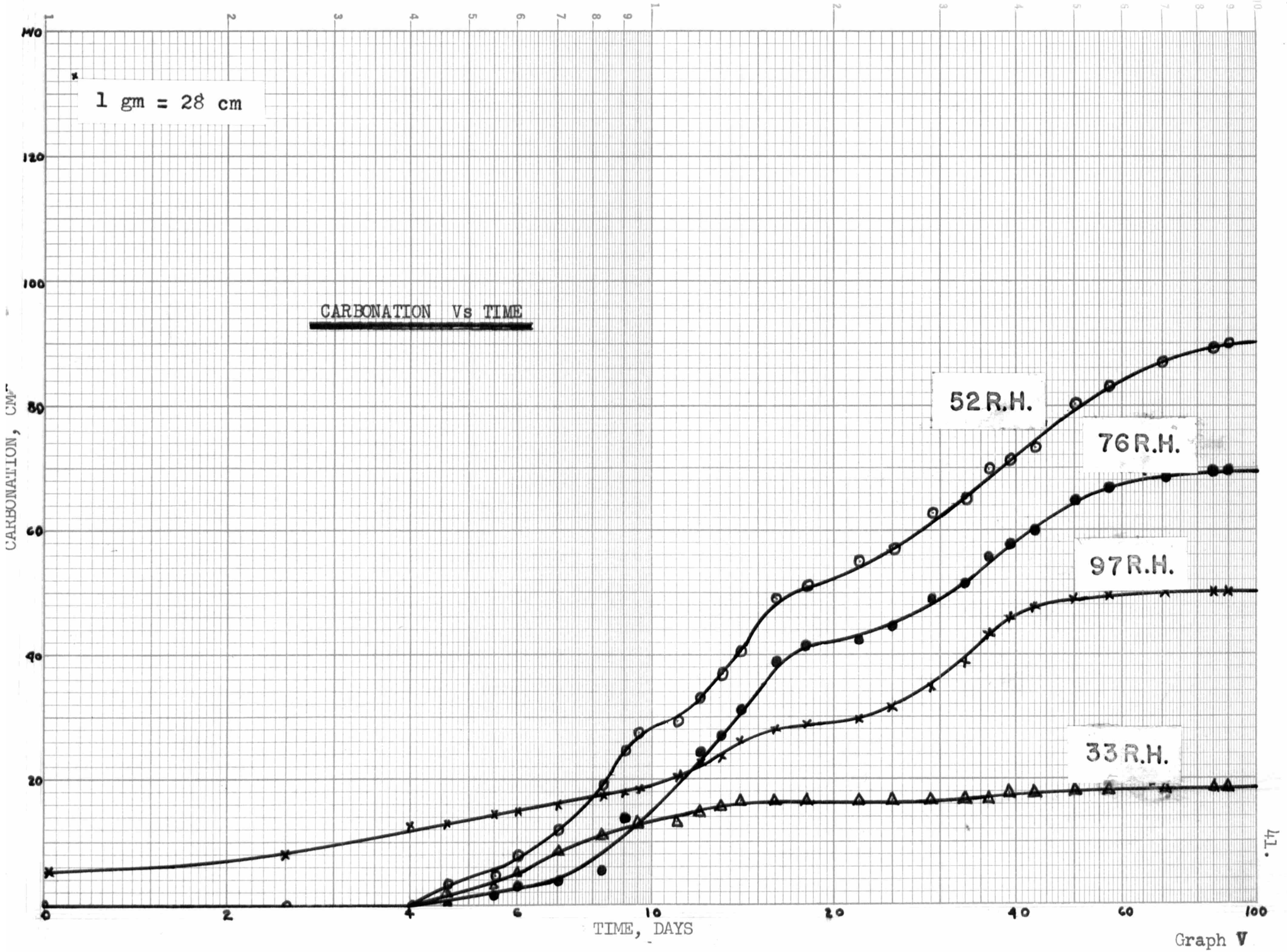

RELATIVE HUMIDITY, PERCENT
100
75
50
25
0
+.02
0
-.04
-.08
-.12
-.16
-.20
-.24
-.28
SHRINKAGE, PERCENT CHANGE IN LENGTH
Shrinkage Due To Carbonation
Shrinkage Due To Carbonation And Creep
Elastic Deformation
Shrinkage Due To Drying
Total Shrinkage
AT: 100 %CO2

Graph IV

TABLE V

CARBONATION VS. TIME

Time (Days)	Carbonation at 97% R.H.	Carbonation at 76% R.H.	Carbonation at 52% R.H.	Carbonation at 33% R.H.
0	0	0	0	0
.4	1.0	0	0	0
1.4	5.7	0	0	0
2.5	8.1	0	0	0
4.0	12.6	.2	0	0
4.6	12.9	.2	3.5	2.5
5.5	14.5	1.9	4.5	4.0
6.0	14.9	2.8	8.0	5.5
7.0	16.2	4.0	12.0	8.5
8.3	17.5	5.8	19.1	11.6
9.0	18.0	14.0	24.5	12.8
9.5	18.2	19.5	27.5	13.2
11.0	20.5	20.9	29.3	13.6
12.0	22.4	24.0	33.1	14.7
13.0	23.9	27.2	37.0	16.0
14.0	26.2	31.0	40.1	16.5
16.0	23.1	39.0	49.1	16.5
18.0	29.0	41.0	51.0	16.5
22.0	29.5	42.4	55.0	16.6
25.0	31.6	44.5	57.0	16.9
29.0	35.1	49.0	62.5	16.9
33.0	38.4	51.8	65.0	17.6
36.0	43.4	55.9	69.9	17.6
39.0	46.2	57.9	71.4	18.0
43.0	47.5	60.1	73.4	18.0
50.0	49.2	65.1	80.0	18.3
57.0	49.7	67.1	83.1	18.3
71.0	50.0	68.5	86.9	18.3
85.0	50.1	69.4	89.2	18.3
90.0	50.1	69.6	90.8	18.3

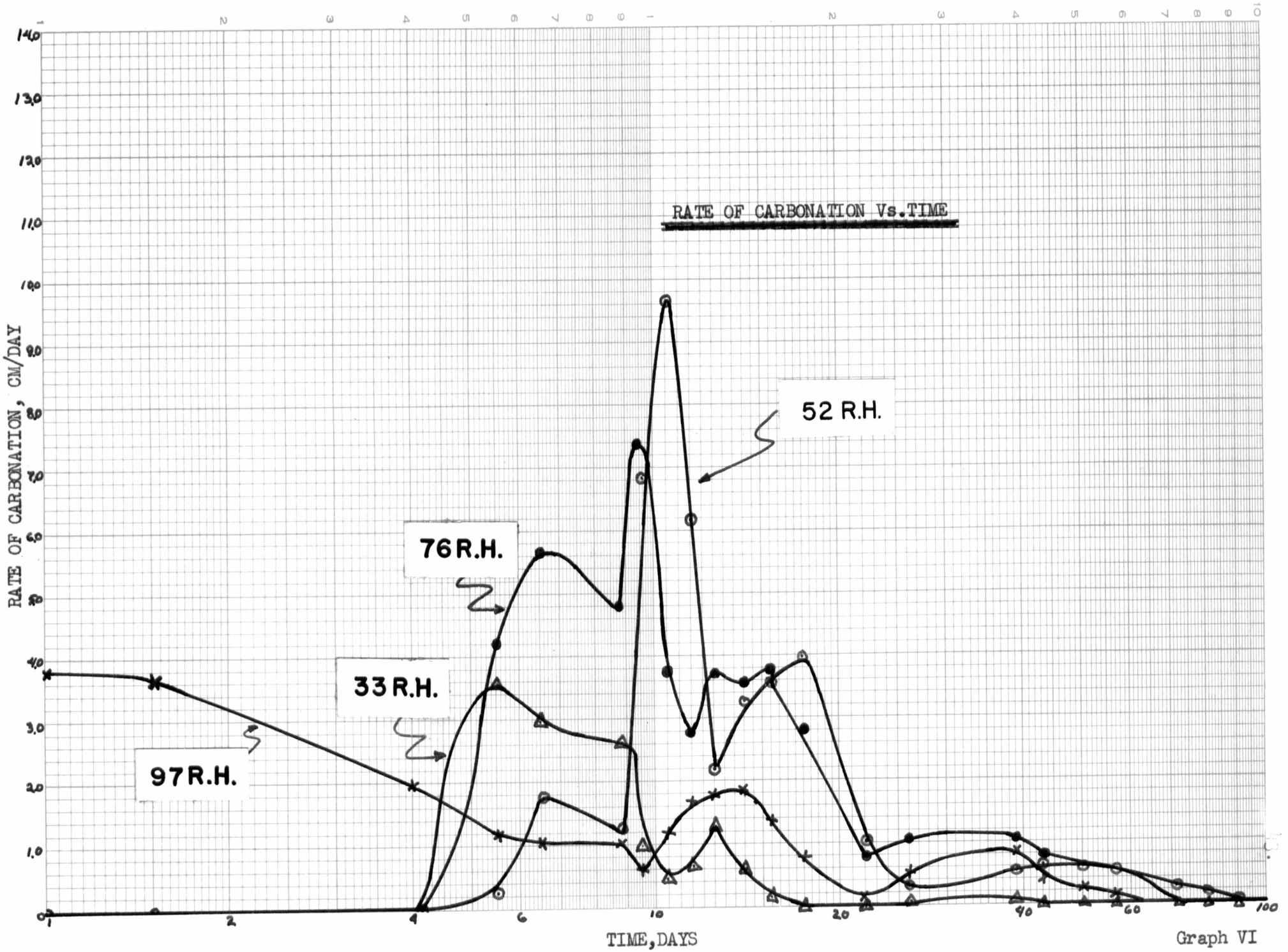

1 gm = 28 cm
CARBONATION Vs TIME
52 R.H.
76 R.H.
97 R.H.
33 R.H.
CARBONATION, CM
TIME, DAYS
0
2
4
6
10
20
40
60
100
120
80
40

Graph V

TABLE VI

RATES OF CARBONATION VS. TIME

Time (Days)	Rate at 97% R.H.	Rate at 76% R.H.	Rate at 52% R.H.	Rate at 33% R.H.
0	0	0	0	0
1.5	3.7	0	0	0
4.0	2.0	0	0	0
5.5	1.2	4.2	.3	3.6
6.5	1.1	5.7	1.8	3.0
7.8	1.1	4.8	1.3	2.7
9.5	0.6	7.5	6.9	1.0
10.5	1.2	3.8	9.7	0.5
11.5	1.7	2.8	6.2	0.7
12.5	1.8	3.8	2.2	1.3
14.0	1.9	3.5	3.3	0.6
15.5	1.4	3.8	3.7	.2
17.0	0.8	2.8	4.0	0
22.0	0.1	0.8	1.0	0
26.0	0.6	1.1	.3	.1
39.0	0.9	1.1	.9	.1
43.0	0.4	.8	.7	0
50.0	.3	.6	.6	0
57.0	.1	.6	.6	0
71.0	0	0	.3	0
80.0	0	0	.2	0
90.0	0	0	.1	0


RATE OF CARBONATION Vs. TIME
RATE OF CARBONATION, CM/DAY
TIME, DAYS
52 R.H.
76 R.H.
33 R.H.
97 R.H.
14.0
13.0
12.0
11.0
10.0
9.0
8.0
7.0
6.0
5.0
4.0
3.0
2.0
1.0
0
1
2
4
6
10
20
40
60
100

Graph VI

IV. DISCUSSION OF RESULTS

Photographic Data

The samples were broken and tested with phenolphtalein which showed alkali reaction on part of the broken side of the specimen but little or no reaction on the outside surface (which had been in contact with the carbonic gas). Thus this test showed that the lime originally present near the surface has suffered a chemical change. It is important to have in mind that the cement gel is ultimately affected by the action of carbonic gas. Therefore the phenolphtalein test should be considered only as a close approximation of what truly is the depth of carbonation.

Photographs I, II, III, and IV show the depth of carbonation of the specimens tested. The carbonation increased toward the center of the sample as the gas diffused inside the sample. Notice the depth of carbonation for the samples maintained at 52 percent relative humidity is markedly increased in comparison with the corresponding sample at the same carbonic gas concentration.

Figure 3 shows the qualitative depth of carbonation observed in the tested samples.

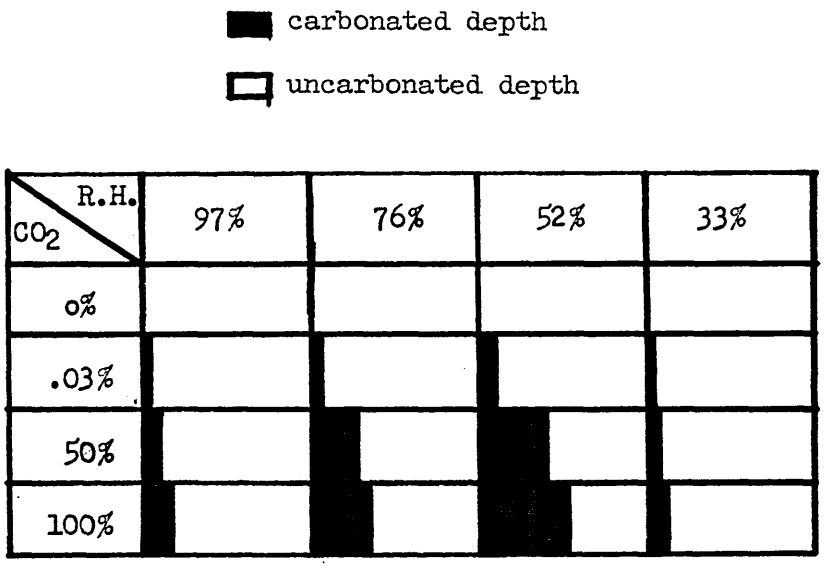


FIGURE 3

Qualitative Depth of Carbonation

Absorption Curves for 50% CO_2

The carbonic gas used during the carbonation period showed that the optimum carbonation occurs at 52% relative humidity for the 50% CO_2 content. The apparent explanation for such action is that the sample in 52% relative humidity contained the optimum water for carbonation on its pores. Therefore the process started as soon as the gas dissolved in the water. Probably the samples that were drier did not contain sufficient water to start such action in a large scale until some water was released by the initial reaction.

Carbonation will proceed until the gas cannot get in contact with the free lime on the sample interior, but only indirectly, by propagation by the water produced by external carbonation. The samples

at higher relative humidities contained large amounts of water in their pores. This water prevented the occurrence of gaseous diffusion of carbonic gas on the sample. Since some calcium ions are counter-diffusing toward the surface it was expected that some calcium carbonate was going to be precipitated. The calcium carbonate will close (clog) the pores preventing carbonation to reach any further stage. It seems that under these environmental conditions the nature of this clogging lies in the formation of $CaCO_3 \cdot 6H_2O$ which is physically bigger than ordinary $CaCO_3$. The initial gas diffusion in the free water on the surface accounts for the early increase in rate of absorption of the wetter samples.

The Rate of Carbonation

The rate of carbonation of the samples (see Graph VI) shows very interesting results. The absorption of CO_2 by the samples was observed to be a maximum during the early stages of the carbonation period. The inference is that most of the carbonation action occurs shortly after (during the first two weeks) the samples are in contact with the atmosphere. The absorption slows down after the saturation of the surface water with carbonic gas. Part of this gas combines with dissolved calcium hydroxide and precipitates calcium carbonate; the rest diffuses inside the sample and continues the reaction until it is used up.

It is interesting to notice too, that the carbonation process started 4 days later for the 76, 52, and 33 percent relative humidities

samples. This was probably due to the absence of excess moisture or large amounts of free water in the surface of these samples. The increased amount of moisture will initially dissolve a larger amount of carbonic gas than would dissolve in samples with these deficiencies.

It is of interest that the rate of absorption of CO_2 decreases (ultimately becomes zero) as a function of time. The apparent explanation is that there is an absence of material susceptible to the action of carbonation, and that the carbonation is dependent upon the permeability of the carbonated depth.

Length and Weight Change at Different Relative Humidities

The graph of relative humidity vs. shrinkage showed that the shrinkage due to water loss increased as the relative humidity decreased. The weight data showed the same trend with respect to weight of the sample, i.e., increased shrinkage decrease weight.

The shrinkage due to carbonation was optimum in the vicinity of the 50% relative humidity. From Graphs I, II, III, and IV the general shape of the carbonation curve was established for different carbonic gas concentrations.

The creep flow of the samples was fairly constant at different relative humidities for low carbonic gas concentrations.

The creep flow seems to be reduced for samples subjected to higher carbonic gas quantity at the optimum relative humidity (50% R.H.). The author suspects that the "sealing action" of the carbonated crust has a bearing on such behavior. A more reasonable explanation

lies in the possibility that the carbonation shrinkage of the loaded specimens was slightly lower due to partly closing of the pores under load.

The elastic deformation upon loading increased for drier samples, i.e., the samples which stabilized at lower relative humidities exhibited larger deformations. This may be explained by the expected decrease in modulus of elasticity of drier samples.

The total shrinkage curves showed that the action of higher concentrations of CO_2 does not necessarily increase the magnitude of the total shrinkage significantly.

V. RECOMMENDATIONS

1. That more extensive analysis should be made of the mechanism of carbonation on shrinkage and creep.

2. Tests should include such variables as water cement ratio, specimen size and carbonic gas concentration.

3. Tests should be run to determine the magnitude and rate of water loss due to simultaneous drying and carbonation.

4. Cement type and methods of curing seem to influence receptiveness and should be investigated fully.

5. The time of testing should be increased to maximize creep deformation.

6. Tests should be made to determine the action of the rate of carbonation for subsequent industry application.

7. The rate of carbonation of samples maintained at 100% carbonic gas concentration should be measured using a similar technique to the one employed on the 50% carbonic gas concentration on this report.

VI. CONCLUSIONS

1. Optimum carbonation shrinkage occurs between 50 and 75 percent relative humidity.

2. The effect of relative humidity is extremely important.

3. There exist no conclusive evidence that carbonation reduces creep.

4. Carbonation shrinkage of loaded samples was reduced due to slight closing of the sample's pores.

5. Increased carbonic gas concentration does not increase carbonation in the same proportion, i.e., double CO_2 concentration does not double the ultimate shrinkage.

6. Carbonation is critical during the first three weeks of exposure to the action of the carbonic gas.

7. There seems to exist correlation between changes in weight and shrinkage deformation.

8. The depth of carbonation increased with increased carbonic gas concentration.

9. Pre-carbonation may prove useful in concrete units of construction if used wisely.

APPENDICES

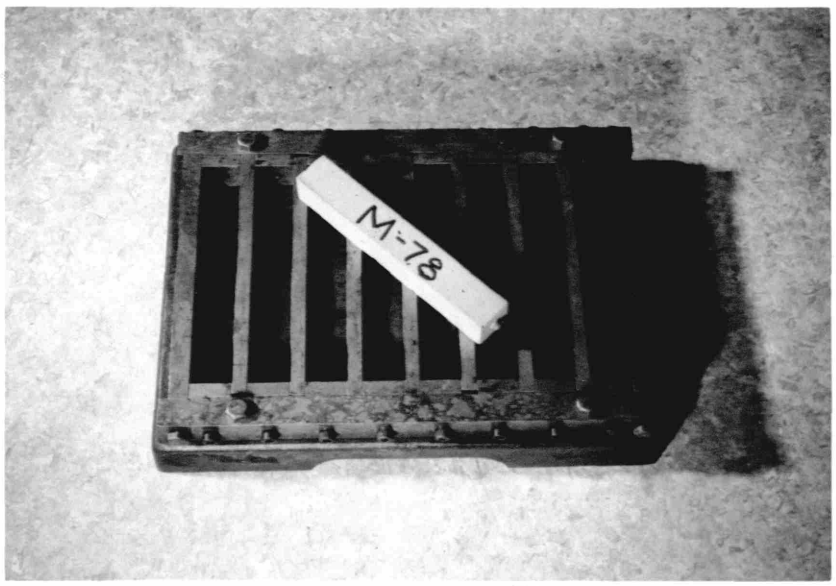
APPENDIX A

Test Procedure

The stages involved are casting, curing, stabilization, loading and carbonating.

Casting

The samples (see Photograph 5) used in this experiment consisted of 1" x 1" x $6\frac{1}{4}$" long mortar prisms prepared in the molds shown.



Photograph 5

SPECIMEN AND MOLD

Plugs were inserted at the end of each sample as reference points to measure shrinkage deformation. Further information with respect to the use of this mold is provided in A.S.T.M. Standard C157-54T.

The mortar was prepared from a blend of four A.S.T.M. Type I Portland cements, locally available (IRON CLAD, DRAGON, ATLAS, and HERCULES). This blend was obtained by mixing equal weights of each brand of cement and mixing throughly with a mason's trowel. The sand used was standard Ottawa sand, well graded.

A mixture of 940 gm. of cement and 470 gm. of water was used giving a water-cement ratio of 0.50 by weight. The mixture was tested for normal consistency according to C109-58. If the mortar displayed a lower consistency, it was disregarded and an increased amount of sand was used repeating the following process of mixing.

The water was placed in the bowl; approximately half of the cement was added and mixed for 30 seconds. The remainder of the cement was added and stirred for 30 seconds. At the end of that time a paste of uniform constitution was obtained. The sand was added to the cement and mixed with irregular movements for two minutes. The normal consistency test followed. Once the normal consistency had been reached (100-110 percent increase in lower diameter of test cone) the amount of sand was recorded.

The final proportions of the mortar were:

240 gm. of cement

470 ml. of water

2,350 gm. of sand

This can be expressed as 1 part water, 2 parts cement and 5 parts sand by weight. The ratio of cement to sand was 1/2.5 by weight.

The specific gravity of the mortar was measured in the laboratory and compared with that calculated from the individual specific gravities of the mortar particles. The difference in the specific gravities was found to be approximately 8%. This shows that the mortar was fairly porous.

The molds were filled and tampered by using a small wooden rod until the cavity was completely filled with mortar. Trowelling was necessary, to assure a uniform surface of the sample, on the top part of the mold. Sixty-four samples were needed for the test.

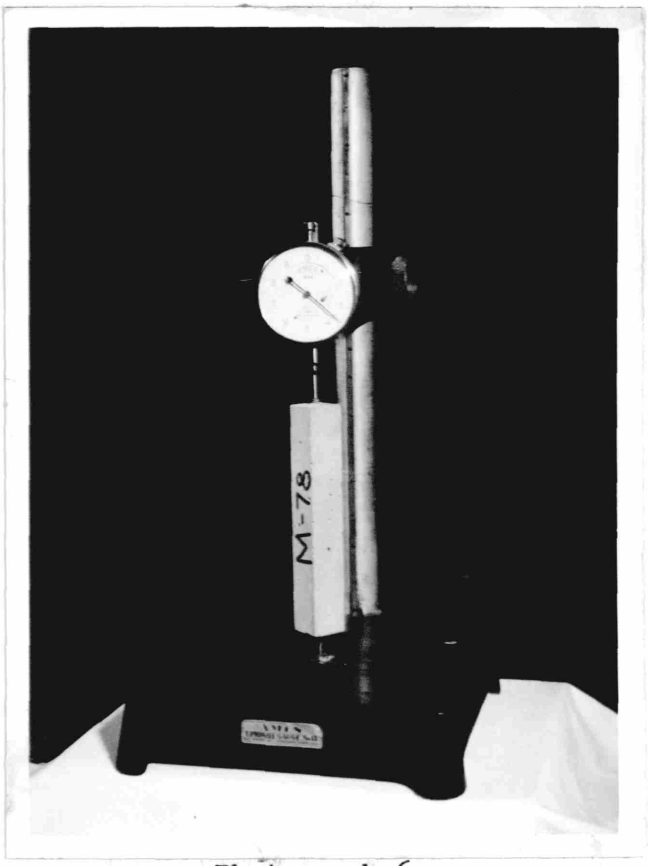
Curing

The molds containing the samples were placed in the moist curing room which had a temperature of 73°F and a 50 percent relative humidity. They were covered with wet burlap. After 24 hours in the molds the specimens were stripped-out and maintained in the same room, and covered with wet burlap for 7 days.

At the end of the curing period the samples were measured and weighed using the equipment shown in Photographs 6 and 7.

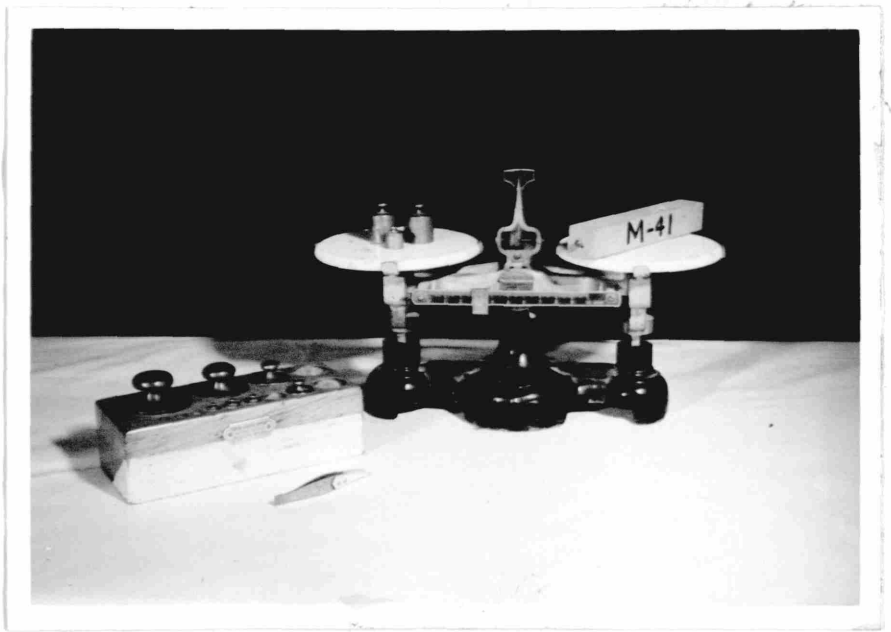
Stabilization

The sample stabilization with respect to weight and length due to water loss was accomplished by using desiccator jars with humidities controlled by saturated solutions of dissolved chemicals. (Refer to Appendix B for further information of these chemicals.)



Photograph 6

MEASURING SAMPLE IN THE UPRIGHT GAGE



Photograph 7

WEIGHING OF SAMPLES

The samples lose moisture until the interior humidity is in equilibrium with the surrounding atmosphere.

The specimens were placed inside the desiccator jars as shown in Photograph 8. Notice that between samples a small wooden stick provides the necessary rollers to prevent any restraining of the samples.

A suction pump was used to remove the air inside the jars and replace it with air free of CO_2. The CO_2 was removed from the air by drawing it through a saturated solution of calcium hydroxide.

Under these conditions the samples were kept for 85 days. At the end of that time it was assumed they were completely stabilized with respect to weight and length.

Once this stage of the project was reached the samples were weighed and measured by the same process used after curing.

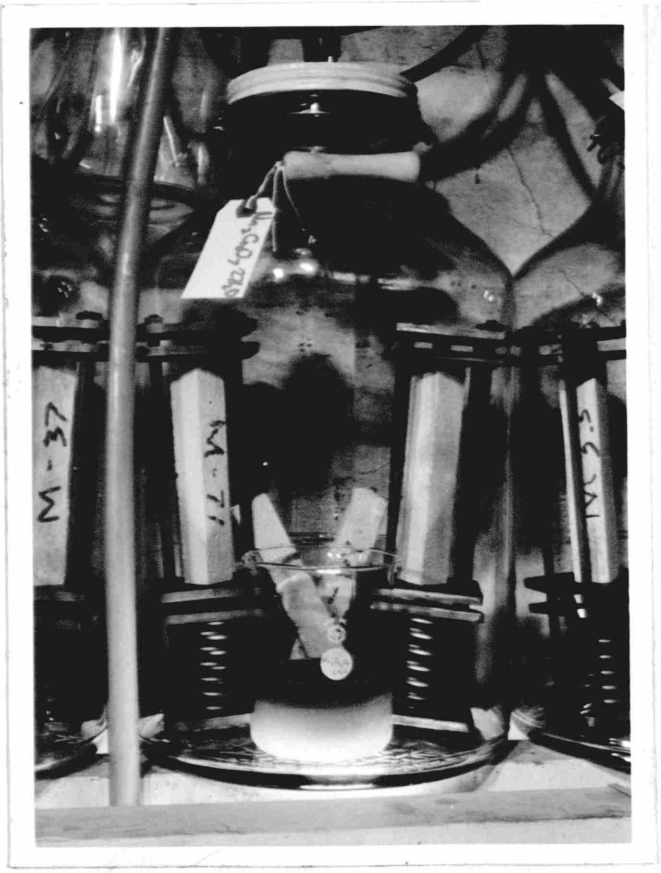
The foregoing procedure assures that subsequent shrinkage and creep will be isolated from initial water loss shrinkage.

Loading

Creep flow as well as shrinkage was considered to be important so half the stabilized samples were subjected to 675 pounds per square inch compressive stress. Companion specimens loaded and unloaded were exposed to 0, .03, 50, and 100 percent carbonic gas concentration at four different relative humidities: 33, 52, 76, and 97.

Photograph 8

STABILIZATION OF SAMPLES



Photograph 9

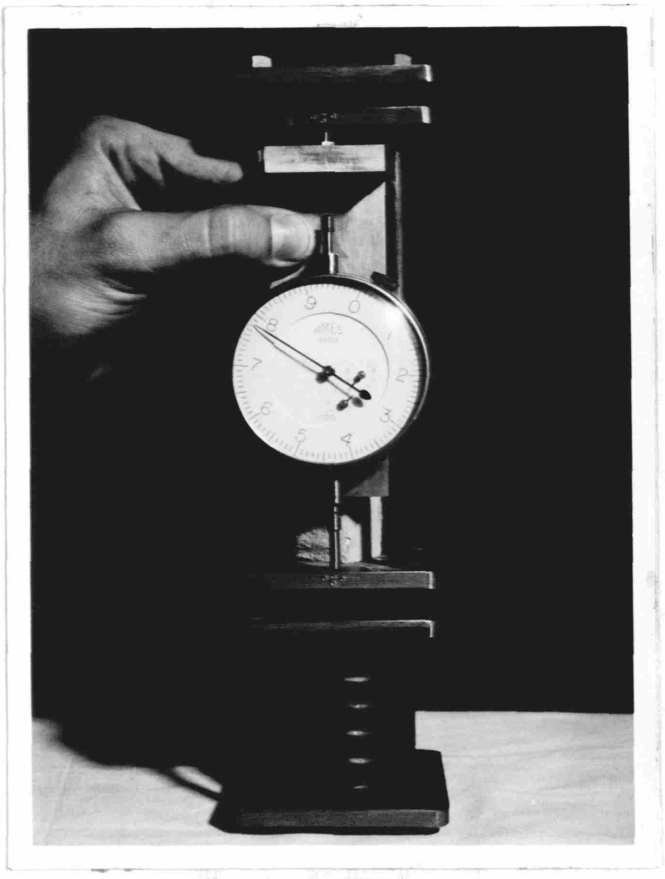
LOADED & UNLOADED

SPECIMENS IN JARS

Photograph 10 shows the attachment used to measure the loaded sample changes in length.

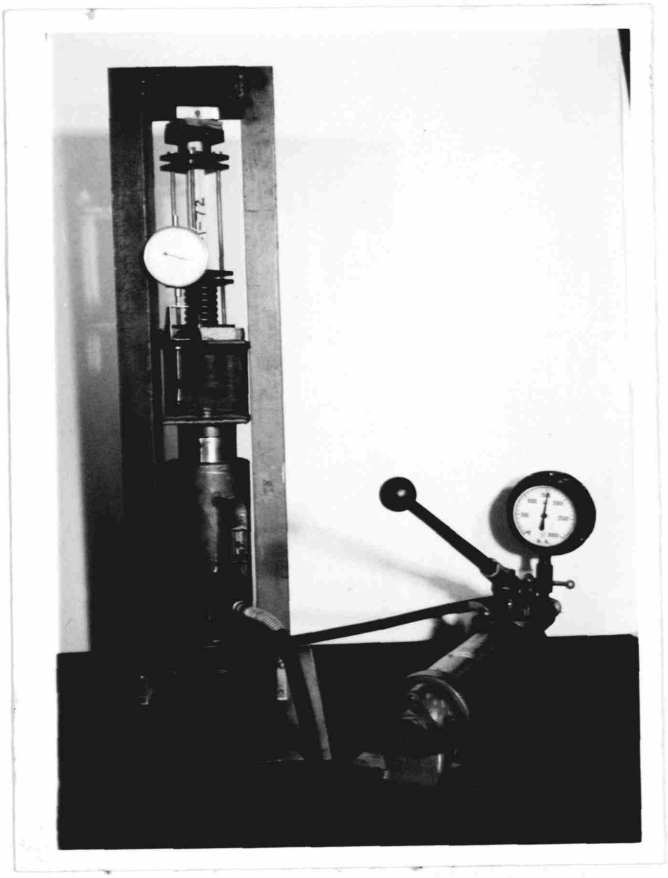
The specimens were loaded by means of the loading frame shown in Photograph 11.

The half inch deformation required on the spring, to develop 675 pounds, was measured by the Ames Dial Gage attached to the lower plate next to the sample and the base of the moving part of the hydraulic jack. Photograph 12 shows the details of the operation. The nuts of the frame bolts were tightened sufficiently to reduce the load applied by the jack to zero.



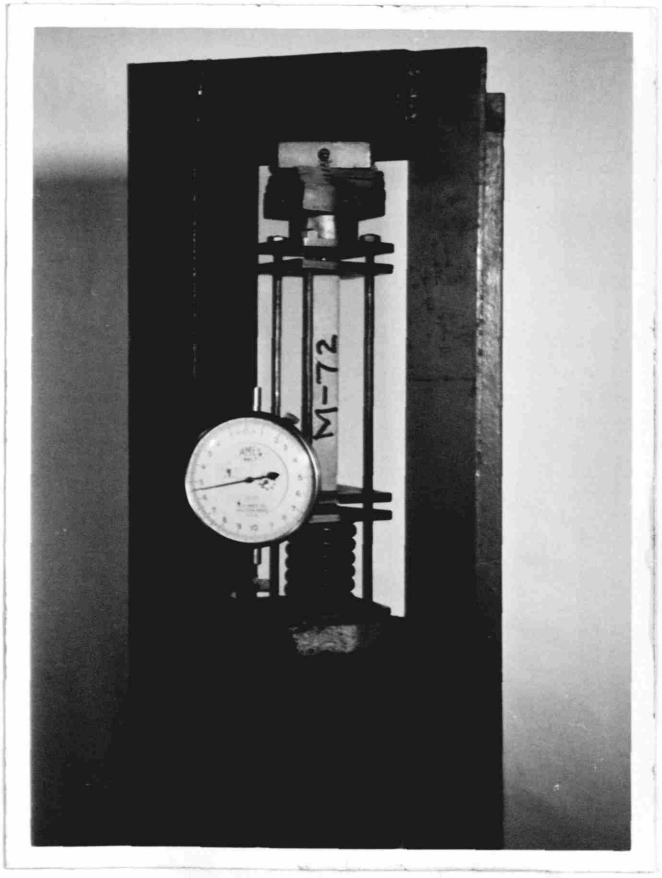
Photograph 10

MEASURING SAMPLE ON LOADING FRAME



Photograph 11

HYDRAULIC COMPRESSION MACHINE AND LOADING FRAME



Photograph 12

MEASURING SPRING DEFORMATION

The shrinkage due to loading was measured by the gage shown in Photograph 10.

Carbonation

After the loading phase the samples were subjected to carbonic gas concentrations of 0, .03, 50, and 100 percent with relative humidities of 33, 52, 76 and 97 percent.

The relative humidities within the jars were maintained by chemical solutions. The carbonic gas concentrations were maintained by different mechanisms as described below.

The zero percent carbonic gas concentration was obtained by the same bubbling method used to obtain CO_2-free air during the stabilization phase. The specimens were sealed in the zero percent CO_2 atmosphere until the end of the carbonation period. See Photograph 13.

Normal carbonic gas concentration was maintained by circulating atmospheric air through the jars using a suction pump working intermittently. See top part of Photograph 14.

A mixed atmosphere consisting of 50 percent carbonic gas and 50 percent air was maintained inside four 5 gallon jars. The detailed procedure for maintaining this atmosphere is explained in Appendix B. Photograph 15 shows some of those jars.

The 100 percent carbonic gas concentration was maintained by the circulation of pure carbonic gas. The lower part of Photograph 14 shows the samples under such treatment.

Photograph 13

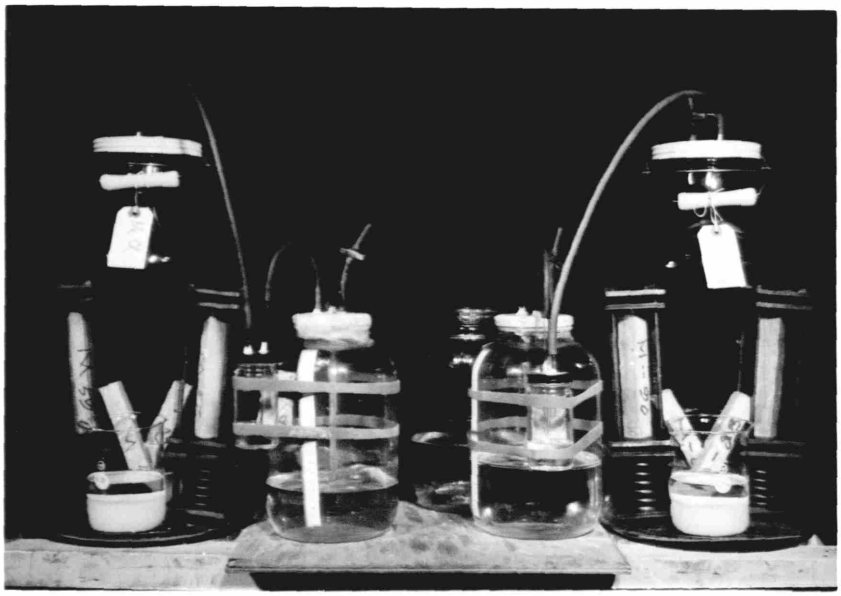
CARBONATION WITH ZERO PERCENT CO_2

AT DIFFERENT RELATIVE HUMIDITIES

Photograph 14

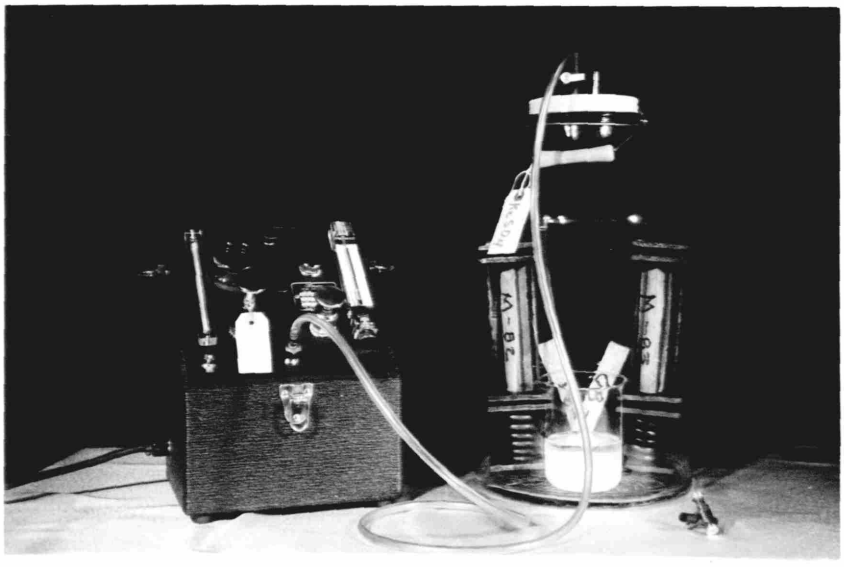
CARBONATION WITH .03 AND 100 PERCENT CO_2

AT DIFFERENT RELATIVE HUMIDITIES



Photograph 15

CARBONATION WITH 50 PERCENT CO_2



Photograph 16

MEASURING RELATIVE HUMIDITY

Appendix B shows the details of construction of all the foregoing equipment.

During the carbonation stage it was desirable to measure the relative humidity of the air inside the carbonation chamber as a check. A "Dew Pointer" humidity gage was used. This gage provided a method of measuring relative humidity without disturbing the atmosphere inside the jar. The principle of its performance consisted of expanding adiabatically a sample of gas, at a definite pressure ratio, until the dew point is attained. By means of tables provided by the manufacturer and a psicrometric chart for different carbonic gas concentrations. The "Dew Pointer" was not used where the carbonic gas concentration was other than as in normal air.

The carbonation period was assumed to last 90 days; at the end of which the samples were removed from the jars. The loaded samples were measured on the frame and the unloaded samples by the "Up-right" gage. The samples were unloaded using the compression machine and weighed.

It was desirable to investigate the depth of carbonation of the samples. For this the samples were cut and drops of phenolphthalein were applied to the newly exposed surface. Phenolphthalein in the presence of an alkali turns a pink color. When the surface is not alkaline in nature the phenolphthalein turns transparent upon contact. A more satisfactory and less fugitive indicator for this purpose was Naphtol Green B, which does not color the carbonated surface, but, instead, stains the inner portion green.

APPENDIX B

Desiccator and Commercial Jars

A constant relative humidity in the atmosphere surrounding the samples was desired. The various relative humidities required were obtained by the use of salt solutions recommended in the American Society for Testing Materials, Proceedings (1936). The saturated solutions of these chemicals in the presence of excess water in the atmosphere will dissolve some of the crystals in the bottom of the container, until equilibrium is attained between the liquid and the vapor above it. Subsequent decrease in relative humidity may cause the solution to give up moisture and precipitate salt crystals.

The saturated solutions of the following salts provided the desired relative humidities at 70°F.

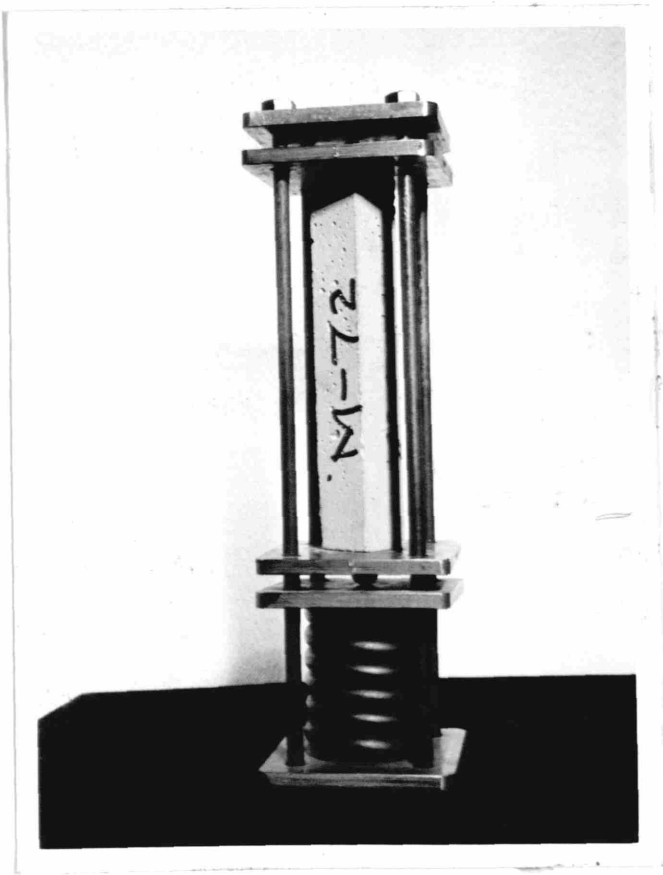
Salt		Relative Humidity (%)
Potassium Sulfate	(K_2SO_4)	97
Sodium Chloride	(NaCl)	76
Sodium Dicromate	($Na_2Cr_2O_7 \cdot 2H_2O$)	52
Magnesium Chloride	($MgCl_2 \cdot 6H_2O$)	33

To maintain the confined atmosphere, the smallest practical conditioning chamber is the ordinary desiccator jar. Desiccator jars are portable and have good ground covers which eliminate leaks and consequent change in salt solutions. They can be placed in constant

temperature rooms or refrigerators. The actual humidity in the desiccator jars was measured by calculating relative humidity from the salt solution used. The commercial jars were sealed with paraffin and the desiccator jars with stop-cock grease.

Loading Frame

The design of a mechanism that could maintain load on the sample while in the jars was built as shown in Photograph 17.



Photograph 17

LOADING FRAME AND SAMPLE

This loading device is rugged, light, efficient and economical. The load is transmitted to the sample by means of a compression spring. Ball bearings are provided to apply the compression load through the longitudinal axis of the sample.

The whole mechanism is based on Hooke's Law. A known distortion will be applied to the spring without exceeding its elastic limit. The spring chosen in the design had a low "spring constant"; the reason for this is small contractions of the sample will not offset the value of the load exerted by more than 1 or 2 percent.

Small marks were filed on the edges of the steel plates next to the sample to serve as reference points from which to take the measurements.

Hydraulic Compression Machine

This mechanism consists of a rigid frame with a hydraulic jack as the load applying unit. The machine was designed to provide a definite deflection on the frame spring and maintain it until the nuts on the loading frame were tightened.

Figure 4 shows some details of the compression machine and loading frame.

Carbonic Gas Concentration Controls

Figures 5, 6, and 7 will show how the 0, .03 and 100 percent carbonic gas concentrations were controlled.

To maintain an equal amount of air and carbonic gas, two jars (with the samples inside) were filled with the two gases. One

jar contained CO_2 and the other atmospheric* air. The gases were mixed by recirculating the contents in a closed circuit by means of a hand pump.

When the samples start using carbonic gas the concentration of it is going to change the original content of 50 percent CO_2 to a lower value. Therefore it is required to replace the amount used. Referring to Figure 8, if a vacuum is created at the jar intake (A) the one way valve aclaims some of the carbonic gas stored to replace that absorbed by the samples. The pressure drops in the CO_2 reservoir causing some water in the second reservoir to flow through the siphon. The water level in the CO_2 reservoir increases. Both levels were maintained at equal heights by adding water at point (B) that was opened to the atmosphere. This will maintain a constant pressure of one atmosphere in the specimens jar. CO_2 can be added to the reservoir by attaching a clamp to point (D) and supplying the gas through (C). Some water will be removed through (B) to account for the increase in volume of gas in the system. The amount and rate of use of CO_2 can be measured by this apparatus.

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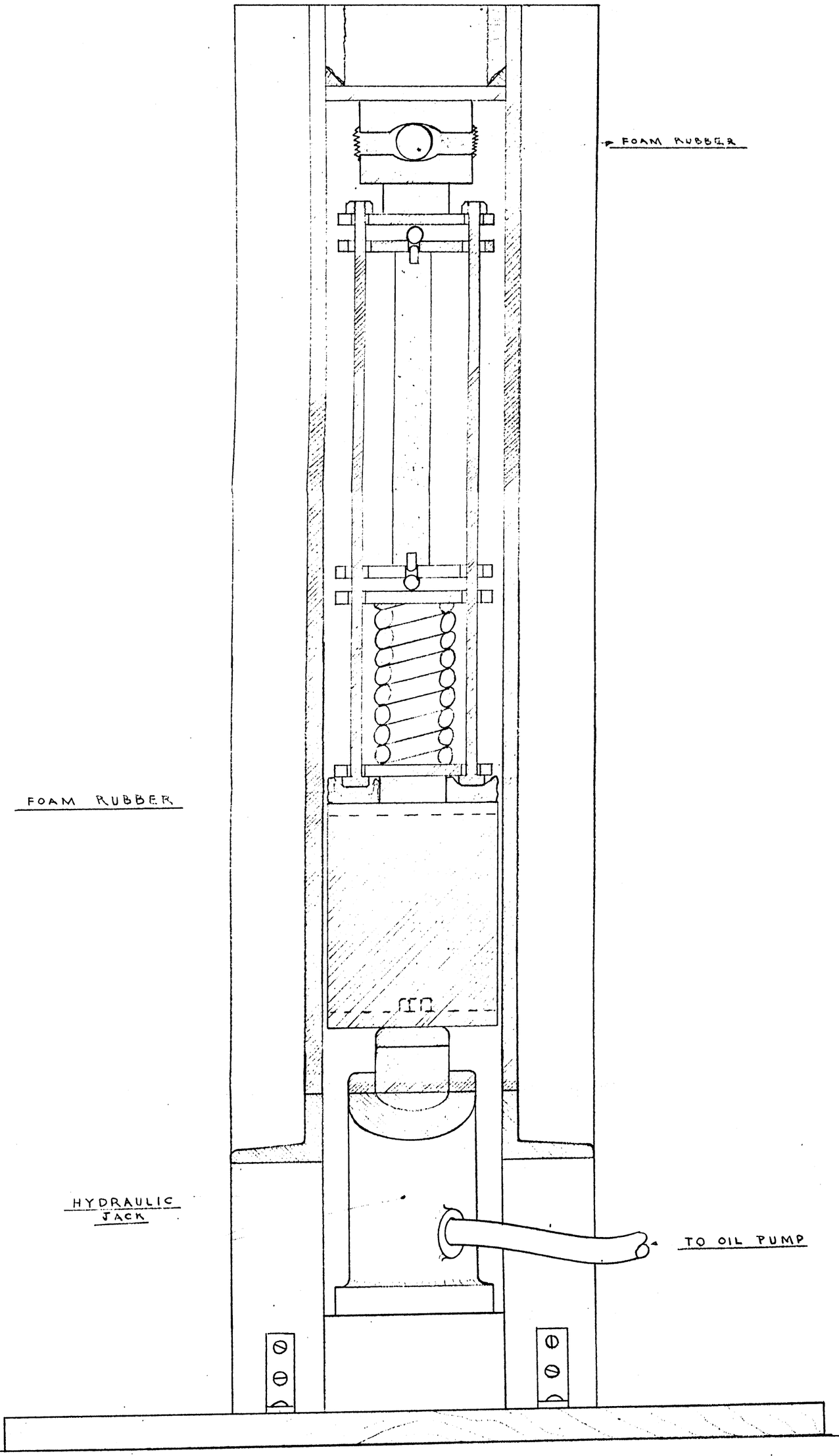
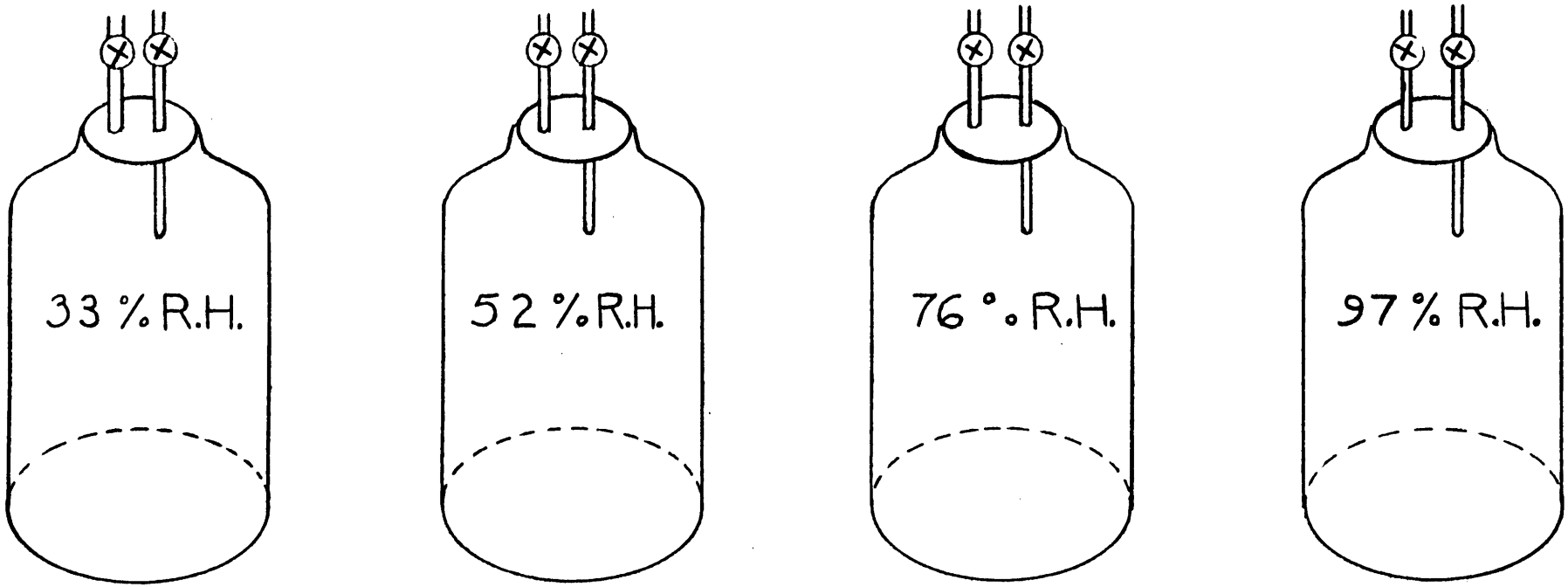
*The .03 percent of CO_2 percent in normal air is neglectable for all practical purposes.



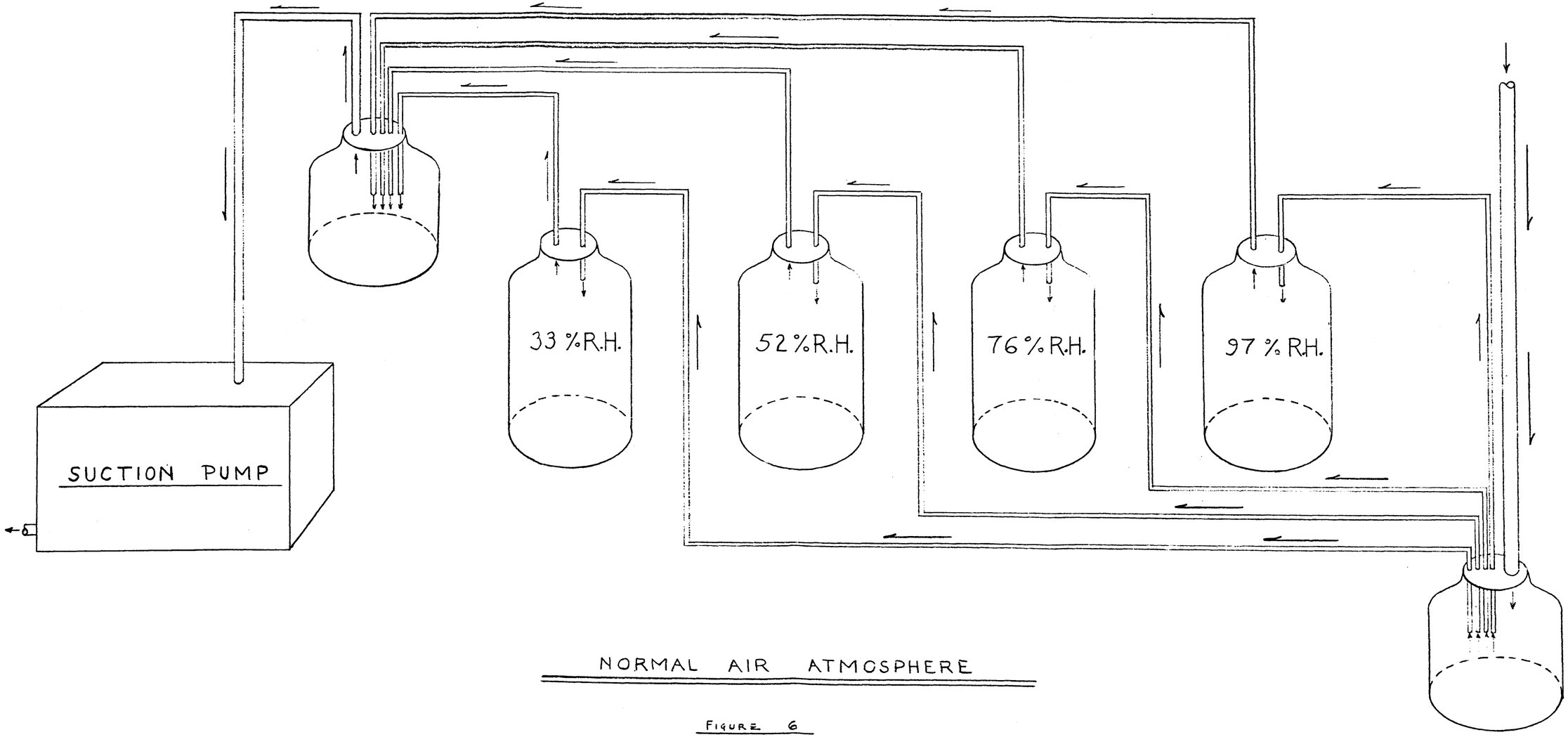
COMPRESION MACHINE AND LOADING FRAME

FIGURE 4



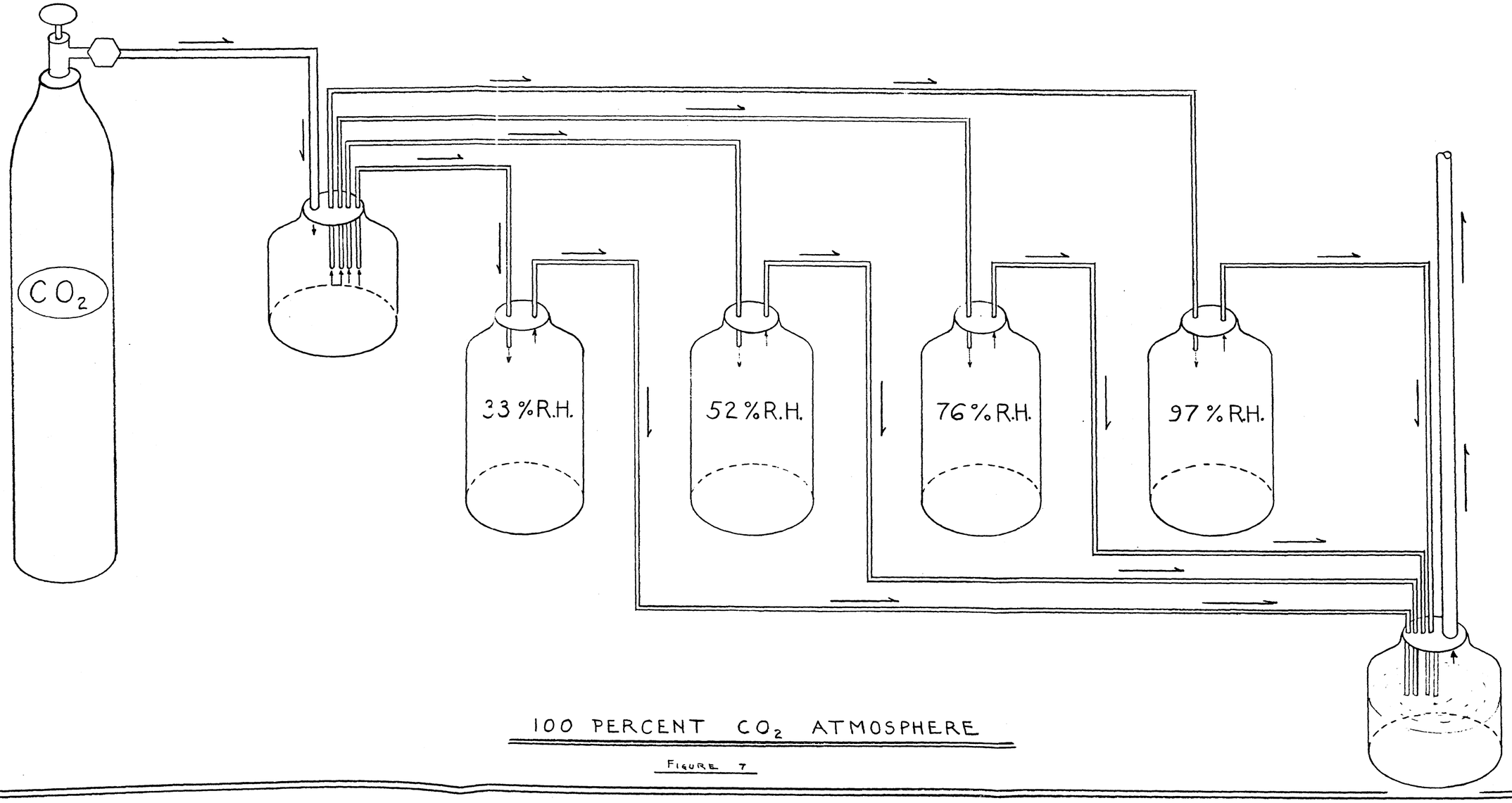
CO_2 FREE ATMOSPHERE

FIGURE 5



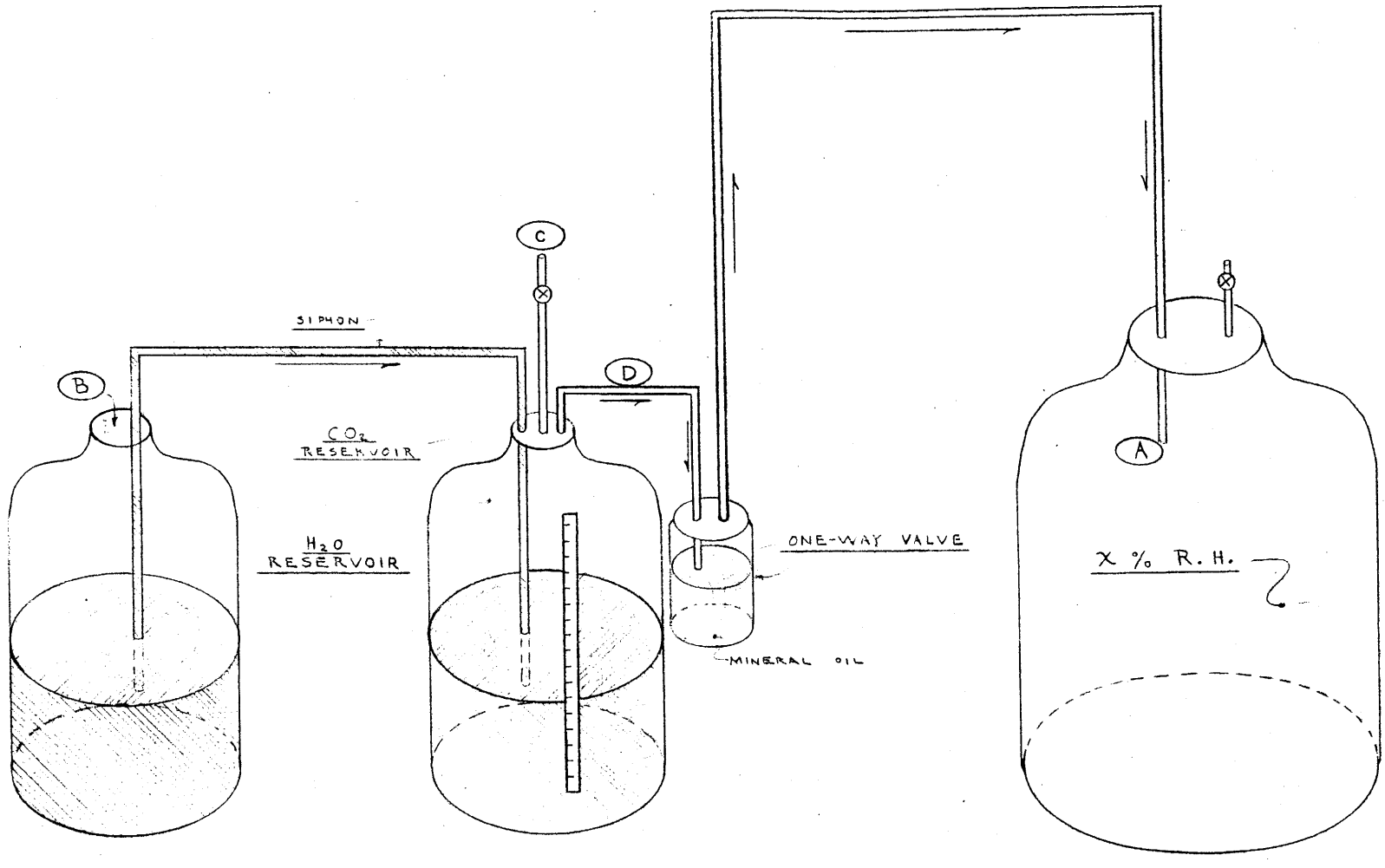
NORMAL AIR ATMOSPHERE

FIGURE 6



100 PERCENT CO_2 ATMOSPHERE

FIGURE 7



50 PERCENT CO_2 ATMOSPHERE

FIGURE 8

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